SULPHUR

The Magazine of World Sulphur Industry

No. 19 *DECEMBER*, 1957

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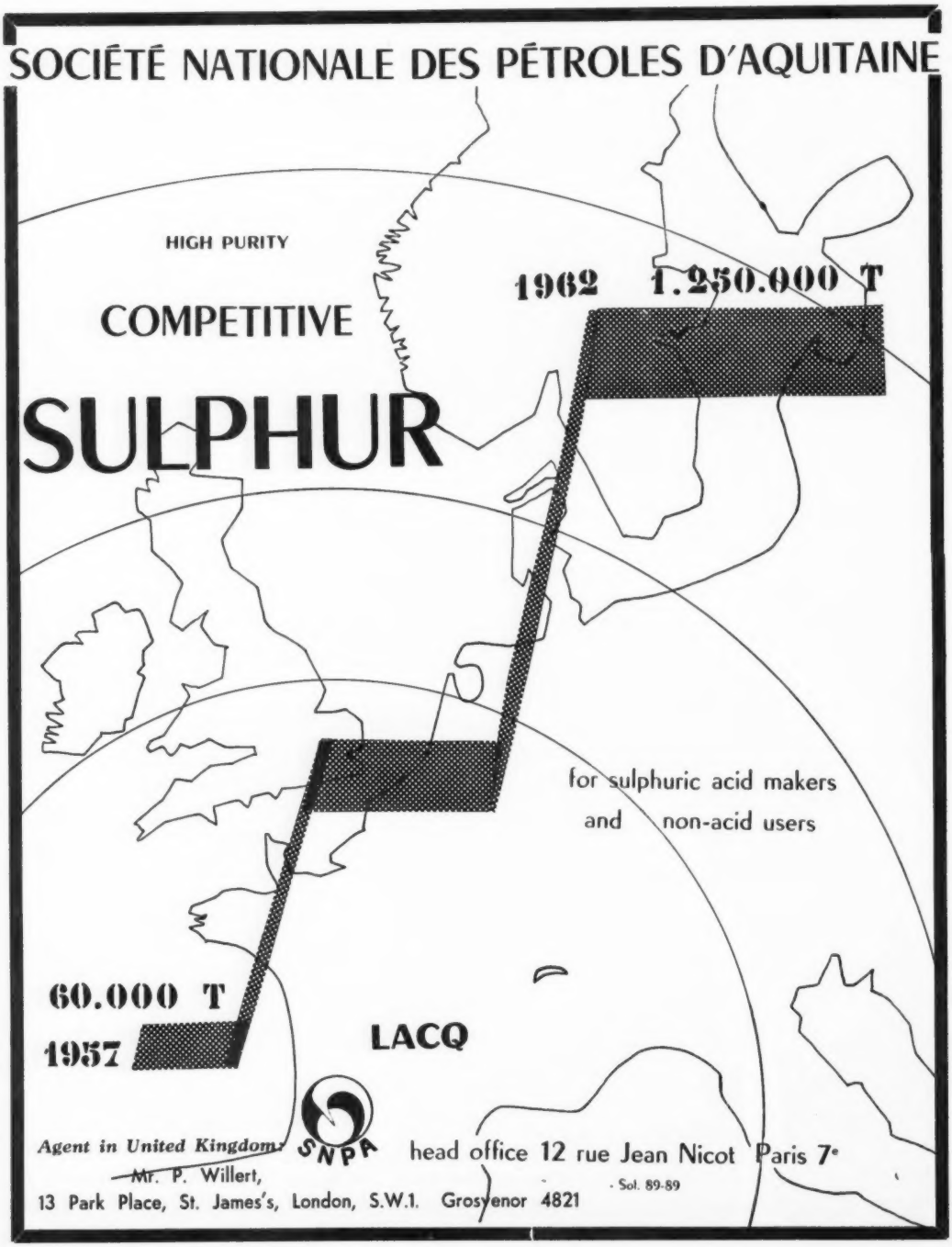
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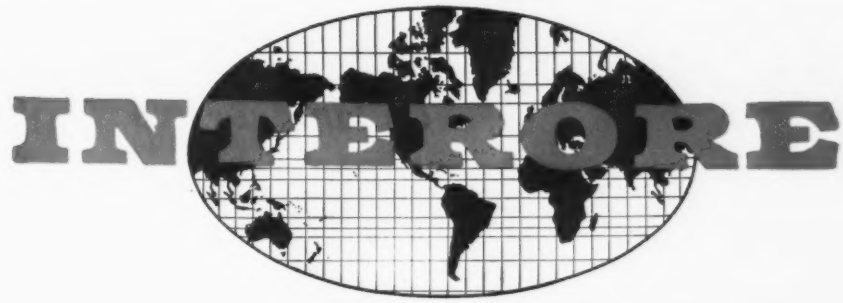

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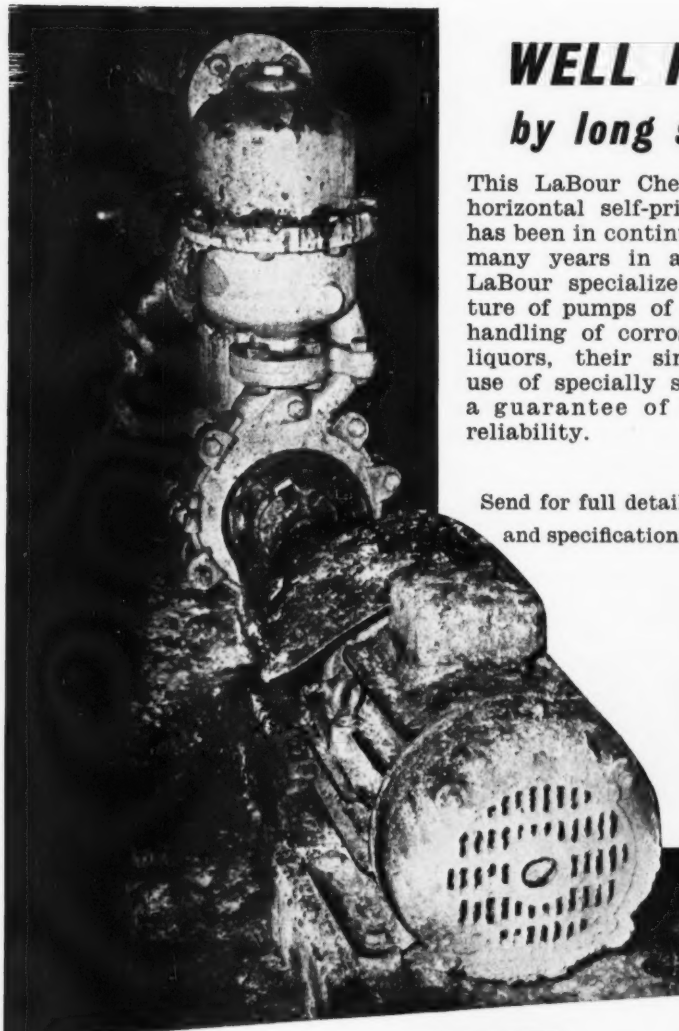

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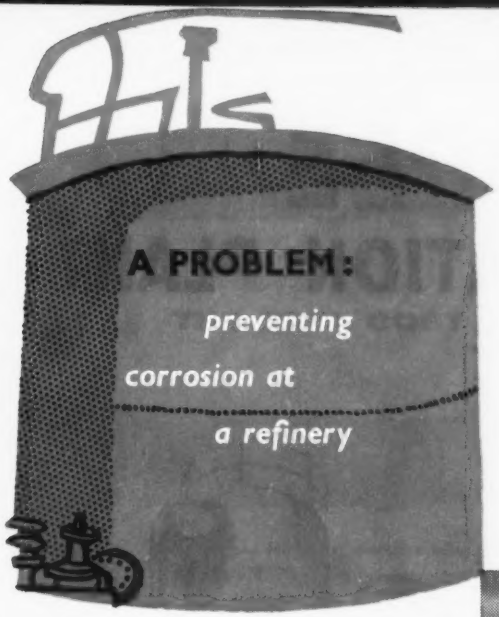

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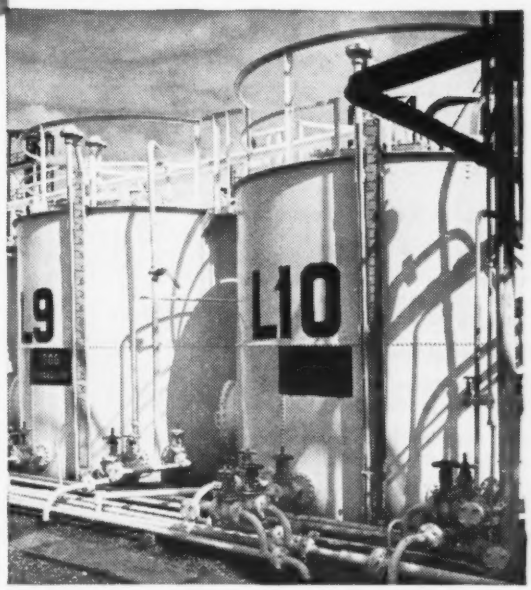

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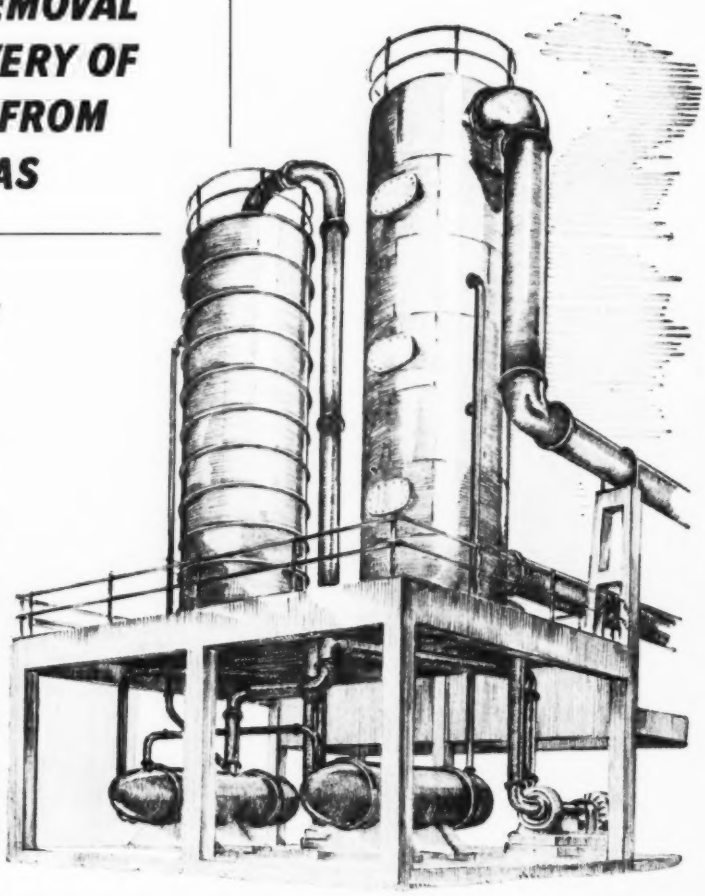

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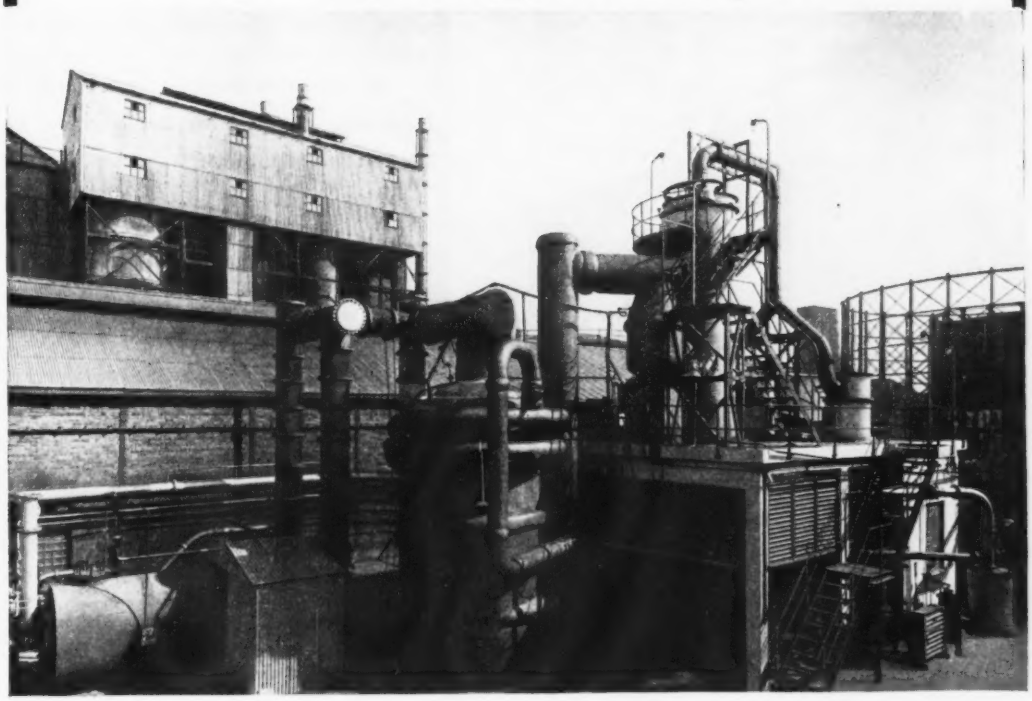

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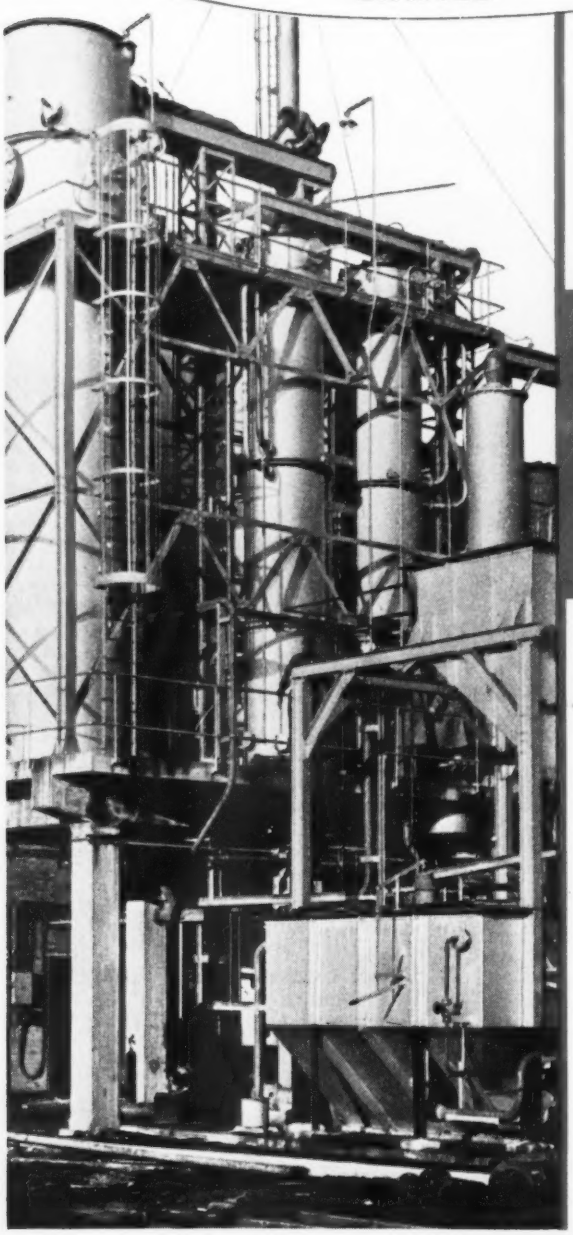

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Review

World prices of brimstone, in terms of U.S. and Mexican supplies, have not recovered stability since the $3 reduction initiated by United States producers in September, and are now $21.50-$25 per ton f.o.b. Gulf, depending on quality and source of supply. Continued availability of shipping at low freights is resulting in exceptionally low delivery costs, in the case of European consumers nearly one-third below the levels of one year ago. Pyrites prices, too, have been reduced following the initiative of the Spanish and Portuguese producers, and prices charged by European mines for delivery after 1st January range from 72s. 6d.-82s. 6d. per metric ton f.o.b.

World supplies of brimstone are ample, and stocks in 1957 are estimated to have increased one-half million to 5½ million tons. The competitive position of pyrites has improved, but heavy consumers' stocks, and slackening demand is expected to prevent mine capacities being fully exploited. The further additions to sulphur dome production capacity in Mexico and major new dome projects in the United States are weakening the statistical position. The main cause of this weakness, however, is the expansion of sulphur recovery facilities and the expected pressure of large-scale brimstone supplies from Canada and France after 1960.

The Sulphur Industry of Canada looks back on 1956 as a year of notable achievement. Production of sulphur in pyrites, by-product acid and recovered brimstone reached all but 800,000 short tons, 22% more than in 1950, to make Canada the seventh largest sulphur producer in the world. The export markets—U.S.A., U.K. and Western Europe—still provided the major stimulus to pyrites production, but the twin sulphuric acid plant project initiated by Noranda Mines Ltd., in the Blind River Uranium field then pointed to the large increase in domestic pyrites use, and in recent weeks this has begun to make itself felt. More than one-half of all domestic sulphur consumption in 1956 of about 870,000 tons, consisted of imported brimstone. The pulp industry is the largest single consumer; next was sulphuric acid for fertiliser manufacture, whilst the acid needs of metallurgical industries, notably for uranium leaching, were growing fast. The outstanding event of the year, however, was the great progress on the Trans-Canada and West Coast Transmission Pipe Lines, as the result of which new brimstone recovery facilities from sour natural gas were being built and planned apace. By the end of 1958 some 800,000 tons brimstone capacity will be completed or under construction, and up to 2½ times more projected for 1960. Already competitive in North Western Canadian and U.S.A. markets and probably the Far East and Australasia, the impact of this growing volume of brimstone on the sulphur industry of Eastern Canada and U.S.A. and on all world supplies will become more strongly felt when as the result of lower rail tariffs their economic radius moves eastwards from Alberta.

The United States Sulphur industry in the third quarter of 1957 produced 1,575,220 tons Frasch and recovered sulphur, the former one-fifth below the record level of the third quarter of 1956. Imports from Mexico at a quarterly rate of 110,000 tons and of domestic recovered sulphur supplies curtailed apparent sales of Frasch sulphur to 1,193,356 tons, 14½% less than in the third quarter 1956. The effect of competition was also apparent in U.S. exports, which were 100,000 tons lower than in the previous year. Lower non-acid sulphur usage in the third quarter, mainly due to the decline in paper, pulp and viscose rayon manufacture, was almost balanced by greater sulphur demand for sulphuric acid production. As the result of increased use in the fertiliser, titanium pigments, oil and uranium industries, acid output rose to 3.94 million short tons 100% H_2SO_4).

The results of the United States' sulphur industry in 1957 are reviewed by **Mr. Langbourne M. Williams,** President of Freeport Sulphur Company. He estimates total sulphur output at 6.9 million tons, including 5.5 million tons Frasch sulphur, whilst United States consumption is estimated at 5.5 million tons and exports at 1.55 million tons. The price reduction of $3 initiated by Texas Gulf Sulphur in September, and followed by all United States sulphur producers, is attributed by Mr. Williams to general competitive condi-

tions, in particular the cheaper offers of Mexican Frasch sulphur.

The **New Dome Developments by the Freeport Sulphur Company** at Lake Pelto and at Grand Isle, six miles off-shore in the Gulf of Mexico, promise to be important on account of their size, whilst new engineering and processing features enhance the reputation of this, the oldest active Frasch sulphur producer. Outstanding and unquestionably determining the economics of both projects, are the use of salt-water as boiler feed, and directional sulphur well drilling, from a single location. The Grand Isle plant is to be built on a Y-shaped steel structure one mile long, 55 feet above the ocean level. Both domes, the company hopes, will be low cost producers.

The Sulphur Industry of Mexico continues developing vigorously. In the third quarter production totalled 245,000 tons brimstone, of which about 96% was in the form of Frasch sulphur. Production facilities of the three active producers, Pan American Sulphur Company, Gulf Sulphur Corporation and Texas Gulf Sulphur Company, have been expanded in recent months, and the level of production has frequently exceeded an annual rate of 1.3 million tons. Texas International Sulphur Company is expected to start producing in the course of this year, while under an agreement in respect of the Sulphur Exploration Company's concessions, Freeport Sulphur Company is now engaging on a major drilling programme. Exports in the third quarter totalled 233,000 tons, of which five-sixth were shipped by Pan American Sulphur Company, the world's third largest brimstone supplier, and the balance by Gulf Sulphur Corporation. The rapid growth of domestic sulphur using industries, notably sulphuric acid, points to the expansion of consumption from the present annual level of 80,000 tons brimstone to one-quarter million tons by 1960.

Sulphur consumption in the United Kingdom during the third quarter totalled about 223,000 tons commensurate with the higher level of general industrial activity. To meet the sulphuric acid demand of 553,738 tons (100% H_2SO_4), unusually high for a summer quarter, production was 5¾% greater than in the corresponding period of 1956, and totalled 545,828 tons (100% H_2SO_4). Greater "acid" sulphur use and the continued high level of anhydrite acid manufacture contrasted with receding pyrites usage and lesser by-product acid supplies caused by the low zinc production. Non-acid sulphur demand stagnated on account of the difficulties of the viscose rayon industry. The competition for a greater share of the United Kingdom markets amongst suppliers of brimstone and pyrites is being accentuated by the expectations of greater availability of domestic recovered sulphur.

Messrs. W. & H. M. Goulding, of Dublin, whose fertiliser manufacturing activities date back over one hundred years, now operate eight plants in Northern Ireland and the Republic of Eire. In the production of sulphuric acid, which takes place at all but one of their plants, some 35,000 tons sulphur in the form of brimstone, pyrites and spent oxide is used. Under the present development programme the number of plants and the level of activities is being expanded to provide adequate supplies of fertilisers especially suited to meet the requirements of Irish agriculture.

The Sulphur Industry of Italy produced 162,030 metric tons fused sulphur in the first eleven months of 1957. At the same time a growing proportion of sulphur ore and concentrates is being consumed directly in sulphuric acid manufacture. The reduction of world prices coupled with the diminishing freight advantage over competing sulphur supplies, notably U.S. and Mexican Frasch sulphur, has imposed a check on the high rate of exports which, aided by Government subsidies, Ente Zolfi Italiani achieved early in 1957. Domestic deliveries of about 108,000 tons and exports of 130,000 tons in the first eleven months substantially exceeded production and led to the reduction of stocks to a level of 200,000 tons, which is almost in keeping with the current level of activities.

Northwest Nitro Chemical Limited, after nearly one year's successful operation at Medicine Hat, Alberta, have emerged as a major supplier of high analysis compound fertiliser to Western Canada and North-West U.S.A. Based on Albertan natural gas and recovered sulphur and phosphate rock from Idaho and employing the versatile St. Gobain Union Chemique Belge phosphoric acid process, the company has the means to meet competitively the needs of the market.

THE SULPHUR INDUSTRY OF CANADA

IN 1956 Canada's sulphur industry entered upon an important new development phase. The past 30 years had seen the progressive growth of the use of sulphur in metal sulphides and, far from being spent, this development received added impetus from the novel large scale use of pyrites in the Noranda process and also from new and planned means for using sulphur dioxide in non-ferrous — especially nickel-smelter gases. The outstanding feature, however, which may be expected to make its full impact upon the world sulphur industry before 1960, has been the rapid progress in providing the means of gathering, cleaning and delivering natural gas and of recovering the extracted hydrogen sulphide in the form of brimstone. The small local sulphur industry which in five years has developed steadily to reach an annual production capacity of 30,000 tons, shared by two companies, is now rapidly growing into an industry of world wide impact under the stimulus of a variety of companies many with international connections in mining, oil, chemicals and sulphur.

Sulphur consumption in 1956 was traditionally dominated by the increased brimstone needs for sulphite pulp manufacture but of greater long term importance were the many sulphuric acid projects, particularly those initiated by Noranda Mines Limited at Blind River, Ontario. Much new acid capacity is intended to serve the needs of the uranium industry which promises to become the largest single acid consumer of Canada by the end of 1958.

PRODUCTION

According to the Preliminary Survey of Sulphur in Canada in 1956 issued by the Department of Mines & Technical Surveys, Ottawa, production of sulphur in all forms in 1956 totalled 798,520 tons* and exceeded that of 1955 by 144,000 tons (22%). The output — the world's seventh largest — is wholly recovered and arises in the form of brimstone and sulphur in by-product pyrites, sulphuric acid, and sulphur dioxide.

Details of production were as follows :—

Sulphur in:	*1956*	*1955*
	tons sulphur	
Smelter gas	221,615	224,457
Pyrite and pyrrhotite shipped	542,121	403,986
Brimstone shipped	34,784	25,976
Total	798,520	654,419

Actual production of recovered brimstone in 1956 totalled 33,464 tons based on natural gas and 3,700 tons from pyrites by the Noranda process. In addition, part of the sulphur content of nickel/copper concentrates is recovered at the nickel refinery of Sherritt Gordon Mines Ltd., at Fort Saskatchewan. Chemo-metallurgical processing yields ammonium sulphate as a by-product at a rate equivalent to about 10,000 tons sulphur annually.

Smelter Gases

The sulphur equivalent of 221,615 short tons recovered in the form of sulphuric acid and liquid sulphur dioxide from the exit gases of non-ferrous metal smelters was only fractionally (1¼%) lower than in 1955. About two-thirds of this tonnage originates at the smelter of Consolidated Mining and Smelting Company at Trail, British Columbia, where the sulphur content of the lead/zinc ores and concentrates is recovered from the roaster gases, as sulphuric acid, for use in fertilizer manufacture.

The smelting operations of the International Nickel Company of Canada Ltd., at Sudbury, Ontario, provide Canadian Industries Limited with the means of manufacturing sulphuric acid and liquid sulphur dioxide. The latter is produced in a plant with a rated capacity of 90,000 tons per annum from the copper ore flash smelter gases containing 70% SO_2. During the year the International Nickel

* All quantities in this Review are expressed in short tons —2,000lb.

Company initiated an important research project in conjunction with Texas Gulf Sulphur Company to determine the economics of elemental sulphur production from the exit gases of a new iron sinter recovery plant. The new smelter project of Eastern Mining and Smelting Company at Chicoutimi was advanced and when in operation it will attract nickel and copper concentrates from mines in eastern Ontario and Quebec and produce 150 tons per day sulphuric acid.

Pyrites

Pyrite and pyrrhotite shipped in 1956 are estimated to have totalled about 1.1 million tons and are reported to have had a sulphur content of about 530,000 tons. In addition the above official statistics include the tonnage of sulphur recovered as sulphuric acid from Barvue zinc concentrates at the Arvida roaster of the Aluminium Company. This is estimated to represent about 12,000 tons sulphur equivalent. The apparent increase in shipments of sulphur in pyrites of about 144,000 tons (34%) compared with 1955 is in excess of expectations. Part is due to greater export demand and it is estimated that at 320,000 tons sulphur in pyrites, the 1956 exports shipments were 80,000 tons greater than in the preceding year. Shipments to the domestic market would thus appear to have totalled about 210,000 tons sulphur in pyrites, 60,000 tons more than last year.

The entire output of pyrite and pyrrhotite concentrates originates from by-product tailings at the concentration plants of copper, lead and zinc mines. On account of low prices, none of the abundant primary pyrite and pyrrhotite mines in Canada are exploited. Mining companies who recovered and shipped pyrite in 1956 were:—

Buchans Mining Co.	Buchans	Newfoundland
Noranda Mines	Noranda	Quebec
Waite Amulet Mines*	Noranda	Quebec
West McDonald Mines*	Noranda	Quebec
Quemont Mining Corp.	Noranda	Quebec
Normetal Mining Corp.	Normetal	Quebec
East Sullivan Mines	Val d'Or	Quebec
Weedon Pyrite & Copper Corporation	Weedon	Quebec
Britannia Mining and Smelting Co.	Britannia Beach	British Columbia

Consolidated Mining & Smelting Company used pyrrhotite tailings in its own acid plant at Kimberley, where, however, owing to fertiliser market difficulties, operations had to be suspended in July 1956 for several months. Noranda Mines Ltd., and its subsidiaries* account for about two-fifths of Canada's by-product pyrite production. In 1956 West McDonald Mines output, which is treated at Waite Amulet concentrator, yielded 235,000 long tons

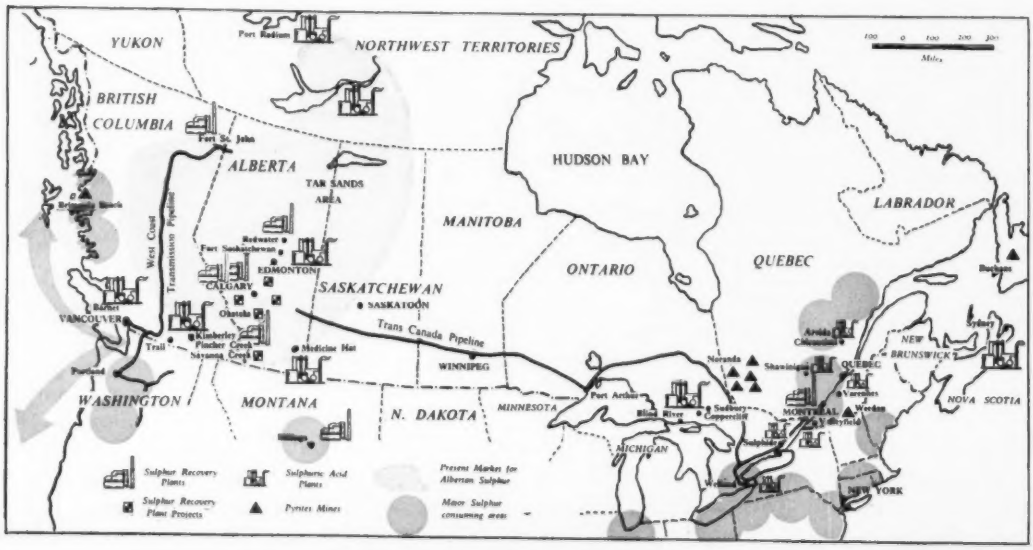


Fig. 1.

pyrites concentrates and after copper and gold extraction from Horne Mine ore some 165,719 long tons pyrite concentrates became available for shipment.

Noranda Process

In 1956—the second year of operation—the Port Robinson plant of Noranda Mines Limited consumed 46,000 tons pyrites concentrates containing about 48-49% sulphur. Processing is reported to have yielded 3,700 tons brimstone and 30,400 tons SO_2 gas which it would seem represents 85% recovery efficiency. The operating rate was at less than one-half capacity due to mechanical difficulties and trouble in producing clean gas. Full scale operations were expected to be possible after April 1957. The new 500 tons per day acid plant at Cutler, which also incorporates the Noranda process, started in July 1956 but initially operated on imported brimstone. Both this plant and a second similar plant are believed since to have come on stream using pyrite concentrates.

Natural Sour Gas

Whereas at the beginning of 1956 only two plants, Shell Oil Company at Jumping Pound and Royalite Oil Company at Turner Valley, Alberta, were in operation with daily capacities of 80 and 30 tons brimstone respectively, the progress on the trans-continental and southward gas trunk lines prompted the rapid promotion of gas treatment and sulphur

British American Oil Co. General view of the gas cleaning installations and 225 tons per day sulphur recovery plant at Pincher Creek, Alberta.

recovery projects. Relevant details are as follows:—

Company	*Location*	*Daily Sulphur Capacity*	*Starting Date*	*Gas Supplies to*
In Operation				
Imperial Oil	Redwater	20	1956	Local Users.
British American Oil Co.	Pincher Creek	225	31/1/56	Trans-Canada Pipe Lines Ltd. and local users.
Jefferson Lake Sulphur Co.	Fort St. John	425	Nov. 1957	Westcoast Transmission Co. Ltd. to British Columbia and U.S.A.
Planned				
Jefferson Lake Sulphur Co.	Fort St. John	375	1959	Westcoast Transmission Co. Ltd. to British Columbia and U.S.A.
.. (with Mobil Oil Co.)	East of Calgary	425	1959	
.. (with Phillips Petroleum Co.)	Savanna Creek	500	1959-60	Pacific N.W. Pipelines.
Group of Local Companies	Calgary area	300	1960	Probably Pacific N.W. Pipelines.
Devon Palmer Oil and Texas Gulf Sulphur Co.	Okotoks	360	Spring 1959	Probably Alberta and Southern Gas Co. Ltd.
Under Consideration				
Provo Gas Producers		No details		
Hudson Bay Oil and Gas Co. Ltd.		No details		
Canadian Fina Oil Ltd.		No details		

Plants in operation and those under construction planned for completion by the end of 1958 are expected to have a combined annual capacity of 800,000 tons high purity brimstone. By 1960 official estimates place output at one million tons, trade circles suggest two million tons installed sulphur recovery capacity as the more likely level of operations.

The H_2S content of the various sour gas fields, which tend to account for an increasing proportion of natural gas supply, range from 2% to 37% which corresponds to about ¾ tons to 15 tons recoverable brimstone per 1 million cubic ft. sour gas throughput.

Sulphur Recovery at Oil Refineries

Initiated in 1956, the project of the Laurentide Chemicals & Sulphur Limited of Montreal is now on point of completion. Gases from five oil refineries and a chemical plant in Montreal East have been gathered for the extraction of H_2S and recovery of brimstone at a daily rate of 100 tons. About 25% of the feed stock originates from the lights ends of the oil refineries production, from which a gas rich in H_2S and with some CO_2 is extracted. The remainder is obtained by scrubbing the fuel gas streams of the refineries. Diethanolamine (DEA) solution from the plant of Laurentide Chemicals is circulated over the absorption towers in the refineries and the foul amine solution is returned for regeneration. Partial combustion and catalysis in a conventional Claus type furnace converts the bulk of H_2S to elemental sulphur while the tail gas containing residual amounts of H_2S is burned to air. Brimstone is stored and despatched in liquid form to local users.

Athabasca Tar Sands

The pioneering work by Royalite Oil Company and Can-Amera Oil Sands Development Company of Calgary is leading to the development of the Athabasca Tar Sands deposit as an economic source of oil supply, following the successful development of the Coulson centrifugue process during the year under review. Early in 1957 the companies announced their intention of building a 20,000 barrels per day processing plant on their property at Mildred Lake, in the Athabasca Valley, 350 miles north of Edmonton. The process entails the recovery of sulphur which at the above throughput is expected to arise at a daily rate of 140 tons. A cautious interest in 1956 by other oil producers has since been followed by intensive negotiations for concessions in the tar sands area.

Trade

Canada in 1956 continued to hold first place amongst world brimstone importers. The monopoly of United States Frasch sulphur supplies in the Canadian market was broken by a shipment of Mexican sulphur to the new

titanium dioxide plant at Varennes, where a small stockpile was being established pending the completion of the sulphuric acid plant.

Details are as follows:—

Imports:	*Tons 1956*	*Tons 1955*
U.S.A.	472,976	373,373
Other Countries	1,141	—
Total	474,117	373,373

The average import price was Can.$28.10 per long ton in 1956 for United States sulphur and Can.$25.40 per long ton for Mexican supplies. Prices at consumers works in Eastern Canada averaged up to $35 per long ton.

The bulk of Canadian sulphur exports is in the form of pyrites. U.S.A. continues as the major outlet accounting for over one-half while the United Kingdom, Netherlands and France, take up the balance. Since 1955 only export values are published; it is estimated that total exports in 1956 amounted to about 700,000 tons pyrites, 13% more than in 1955, and containing about 320,000 tons sulphur.

Details are as follows:—

Pyrites Exports:	$	$
U.S.A.	1,370,419	1,293,373
U.K.	568,970	458,202
Netherlands	422,970	210,000
France	286,990	40,000
Total	2,649,349	2,001,575

Export prices range from 7 to 12 cents per long ton unit of sulphur and the average value of pyrites is estimated at $4.60 per long ton pyrites. Other sulphur exports totalled 4,331 tons, valued at average $33 per long ton, and were directed mainly to India, but also to U.S.A. and Indonesia.

CONSUMPTION

It is estimated that apparent consumption by Canadian industry in 1956 amounted to 950,000 tons, an increase of about 165,000 tons over 1955. Based on the activities of the various sectors of the sulphur industry, however, sulphur consumption in all forms appears to have been about 870,000 tons, sulphite pulp manufacture taking up 450,000 tons, sulphuric acid manufacture for fertiliser and other uses 400,000 tons and non-acid brimstone uses, mainly carbon disulphide production, of about 20,000-22,000 tons. Imported brimstone totalling 474,000 tons continues to represent the largest single source of supply and, augmented by domestic recovered material, brimstone supplies at the disposal of industry totalled 512,000 tons. The following table shows the latest official details of elemental sulphur usage:—

	1955	*1954*
	tons	
Pulp & paper	300,825	268,607
Heavy chemicals	82,947	80,871
Rubber	2,784	2,360
Miscellaneous	6,573	7,115
Total	393,129	358,953

Sulphite pulp and sulphuric acid manufacture accounted for the bulk of greater requirements in 1956.

Sulphuric Acid Production

Annual production of sulphuric acid in 1956 exceeded for the first time the one million tons mark. Stimulated by increased activity plant capacities were expanded at established

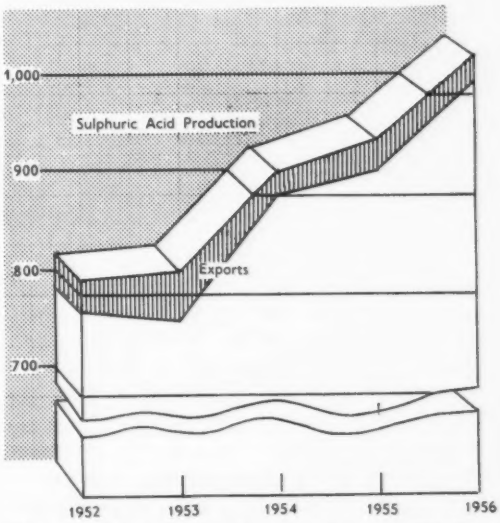


Fig. 2 Sulphuric acid production and exports 1952-1956 in thousand short tons 100% H_2SO_4.

In 1956 net exports are shown; this allows for an adjustment of 2,055 tons imports.

plants and new plants entered production. In the Spring of the year the Valleyfield plant of Nichols Chemical Co., was modified to allow it to make full use of the installed pyrite roasting capacity; its output capacity is estimated to

have risen in consequence by 60% to 160,000 tons (100% H_2SO_4).

Shawinigan Chemicals Limited brought on stream in October a new contact acid plant based on brimstone use with a daily capacity of 75 tons monohydrate in the form of 93% and 98% acid. Built by the Chemical Construction Corporation of New York, it is the Company's only acid installation and supplies the acid needs of the Company's main works at Shawinigan Falls and part of the local market.

Other acid projects which were completed in the autumn of 1956 include Inland Chemicals Limited, whose 100 tons per day brimstone

Northwest Nitro Chemicals Ltd. The new 400 tons per day sulphuric acid plant.

contact acid plant at Fort Saskatchewan supplies the needs of the adjacent nickel refinery and the installation of Northwest Nitro Chemicals Limited at Medicine Hat.

The expansion of the Uranium industry* stimulated development and in addition to the project of Noranda Mines Ltd., at Cutler, Ontario, substantial new capacity was put in hand in the Beaverlodge area where Gunnar Mines Ltd. initiated a new 65 tons per day contact acid project and also among acid makers who are serving the uranium mills in Ontario. Construction also started on a 150 tons per day Leonard/Monsanto contact acid plant at Varennes, Quebec, where Canadian Titanium Pigments Limited are establishing Canada's first titanium dioxide installation.

Sulphuric Acid Usage

Despite the slight curtailment of fertiliser activities by Consolidated Mining and Smelting Co. Ltd., Canada's largest single sulphuric acid user, there was an overall increase of acid consumption for fertiliser manufacture which now represents about 60% of total usage, compared with about 61½% in 1955 and 65½% in 1954. Heavy chemicals, notably aluminium sulphate and hydrochloric acid, the iron and steel industry and oil refineries accounted for most of the increased acid consumption, and significant advances were also recorded in acid used in metallurgy and transparent cellulose films.

The distribution of acid usage in 1954 and 1955 was as follows:--

	1955 tons 100% H_2SO_4	*1954*
Fertilizers	577,100	603,200
Heavy chemicals	139,700	123,400
Non-ferrous smelters & refiners	24,500	19,300
Coke and gas	37,900	31,000
Petroleum refining	6,500	5,400
Leather tanning	2,300	1,900
Iron and steel	35,300	25,800
Electrical apparatus	8,100	6,000
Plastics	15,000	13,700
Soaps and washing compounds	11,300	10,600
Sugar refining	300	300
Pulp and Paper	8,700	9,300
Vegetable oils	100	100
Adhesives	200	500
Miscellaneous	70,800	72,000
Total	937,800	922,500

Conclusion

By an 18% increase to nearly 800,000 tons Canada's production of sulphur in 1956 had nearly doubled in the past five years. Much of this additional output went to expand Canada's sulphur exports which in the form of by-product pyrites have risen to 320,000 tons sulphur content. Domestic consumers, particularly the pulp industry, the largest single sulphur user, were still dependent on imported brimstone for over one-half of their requirements estimated at about 870,000 tons sulphur, almost twice the tonnage used five years earlier.

Although market conditions adversely affected progress in the fertiliser and especially the pulp industry in 1957, the immediate outlook for the continued rise in sulphuric acid use is favourable. This is based principally on the rapidly growing uranium industry and also the needs of other industries notably pigments,

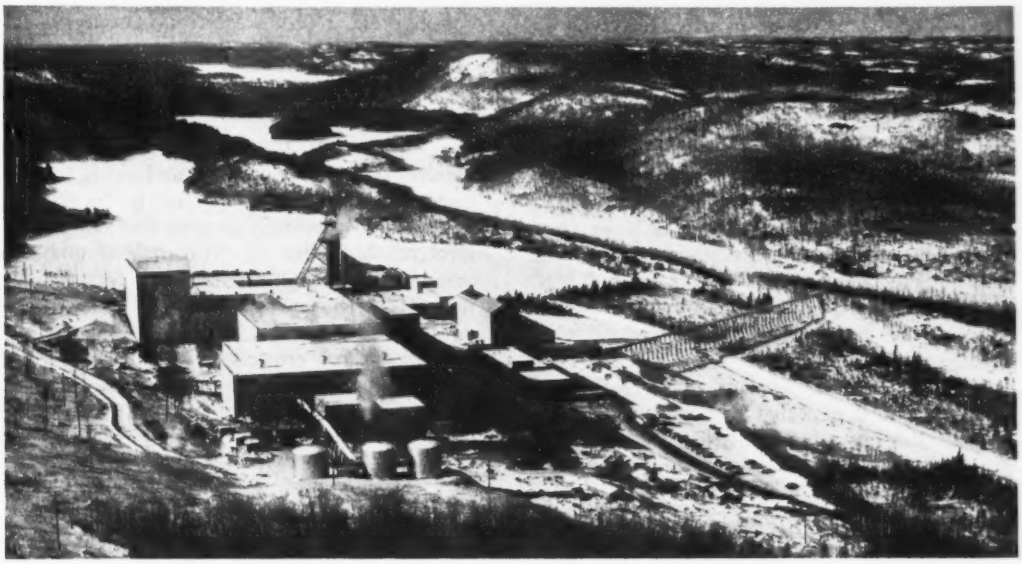
* For full details see Issue No. 18, September, 1957

chemicals, oil and steel which are developing and expanding in the course of Canada's economy's broadening base. In Eastern Canada, Mexican sulphur supplies have challenged the monopoly of the United States Frasch sulphur amongst acid sulphur users and it is expected that in 1958 they will do so in the pulp industry. Simultaneously but depending on the economics of operation of the £20 million twin p'ants built by Noranda Mines Limited at Cutler, Ontario, by-product pyrites supplies may make a serious challenge in the acid sector of the industry.

Dominating the outlook for the Canadian sulphur industry is the rapid progress in providing the means of exploiting the wealth of natural gas in Western Canada. Developments to date have outlined the pattern of imminent growth of sulphur recovery and the variety of operating companies concerned emphasise their potential importance to Canadian and world sulphur markets. The expanding use of cheap gas energy will have as its corollary a mounting volume of sulphur output which, although capable of temporary accomodation in the shape of stockpiles, will inevitably have to seek an outlet.

Already brimstone recovery from natural gas in Western Canada exceeds an annual rate of 100,000 tons and in a matter of months its volume will be such that the present markets of primary sulphur producers in Northwest Canada and the Northwest United States will be seriously threatened. British American Oil Co., at present the largest single producer of brimstone in the Commonwealth is, in addition to local sales, said to be establishing a stockpile in Vancouver with a view to entering the export market in the Far East and Australasia.

The level of sulphur recovery in Western Canada may be expected to reach over two million tons by 1960, a tonnage much in excess of likely local outlets and export markets competitively accessible from Vancouver. The obstacle to supplying the major markets in Eastern Canada and the U.S.A. is the cost of transport and as the railway companies are ready to negotiate lower tariffs to attract a greater volume of freight so the economic radius of Western Canada's recovered sulphur may be expected to advance eastward.



Algom Uranium Mines Ltd. General view of Lake Nordic mine which has a daily milling capacity of 3,000 tons uranium ore.

PRICES and TRENDS

WORLD prices of brimstone during the past three months lacked stability and although there were few actual changes, producers and consumers evinced a lack of confidence in the new price structure. This was manifested by the absence of any exceptional increase in shipments which, as sea freights generally remained at low levels, might have occurred had there been any sign of the market becoming firmer. On the contrary, lower industrial activity in the United States coupled with rising stocks and continuous progress on major new projects underlined the world-wide weakness of the brimstone market.

In the United States all producers base their prices for deliveries to domestic and export markets on $25 per ton f.o.b. or f.a.s. for bright sulphur with discounts of $1 in the case of Freeport Sulphur Company and U.S. cents 50 for Texas Gulf Sulphur off-colour sulphur. As the result of the $3 reduction in price on September 18th 1957 the loss of revenue in the last quarter must have been heavy and it is expected that end-year results will disclose an adverse picture, especially as earlier in the year profit margins had started to narrow alarmingly.

Since the ineffective attempt to reduce the price differential of Mexican sulphur producers, opinion in the United States sulphur industry has ranged from the extreme of further price cuts, if necessary below cost levels, to the demand for Government controls to keep cheap imports out and the domestic price of United States brimstone at an economic level. So far, however, the Frasch sulphur industry — U.S. and Mexican — has not seen its way to giving open consideration to the merits of co-ordinating its production and marketing problems, and the involved anti-trust legislation appears to be far too strong a deterrent to permit responsible leaders of the industry to take any initiative in this matter. Mr. Eugene H. Walet, Junior, President of Jefferson Lake Sulphur Company, at the combined meeting of the Commercial Chemical Development Association and the Chemical Market Research Association held at Houston, Texas, at the end of November, stated a case for import control of brimstone. In his support he quoted President Eisenhower's advocacy of co-operation and self-restraint to control voluntarily the level of crude oil imports into the United Stated. Compared with oil, sulphur in the U.S.A. does not occur as plentifully in commercial quantities and in Mr. Walet's view warrants at least as much consideration. If dome exploration is to continue and reserves are to be developed and fully exploited, he thinks it desirable to encourage and even ensure stable prices for domestic sulphur production.

The recent price reduction Mr. Walet believes to have been engineered solely to meet competition from Mexico and to be without justification, especially as even the $30 per ton level reached after the Korean War does not match the price increases of many other minerals. Reviewing some of the recent developments, notably Freeport Sulphur Company's off-shore projects, Mr. Walet referred to the very high capital cost of creating employment in the Frasch sulphur industry. This amounted to $100,000 per capita compared with average $14,000 for all industry.

Prices of Mexican Frasch sulphur which had receded in September and early October to re-establish the earlier differential $2-3 below the U.S. price level were modified on 8th December. Both Pan American Sulphur Company and Gulf Sulphur Corporation announced that future sales of fully-filtered material would be based on U.S. $24 per ton f.o.b. Coatzacoalcos, $1 above earlier quotations. This may be taken as a conciliatory gesture to the major U.S. producers notably Texas Gulf Sulphur but its effective value is not expected to be significant, as prices to established customers of the Mexican producers are not affected and as the issue concerns primarily the U.S. market where the low price of crude and semi-filtered material coupled with the efficient sales service, out of stockpiles established at Tampa and on the Eastern Seaboard, are often the main attraction.

The Italian sulphur industry has been severely embarrassed by the lower level of world prices; the necessity to reduce its prices

by the greater application of subsidy is causing a severe drain on the limited resources of Ente Zolfi Italiani. The notional exports sales price remains based on L.23,000 per metric ton (£13 7s. per ton) for Buona Sicilia (min. 98% S). It appears that competitive sales in Mediterranean markets require an additional subsidy of at least L.5,000 — equal to about £3 per ton — and a little less in the case of sales to Balkan and Central European countries where the freight advantage over Mexican and U.S. supplies favours Italian sulphur.

In Japan rising costs contribute to the difficulties of the sulphur producers who are facing a demand for lower prices as the result of growing domestic recovered sulphur production and the prospect of cheap imports from Canada. Only the Government import ban and the support of the status quo appears to have prevented a break of the price agreement between producers and consumers which established, in 1956, the domestic sulphur price at Yen 22,000 per metric ton f.o.b. mine.

China has recently placed elemental sulphur, refined from native ore, on offer through merchants in United Kingdom and Western Europe at £12 per ton f.o.b. Tientsin. The material is reported to be high in Arsenic (about 0.005%) and in Selenium (6-8 p.p.m.), but the sulphur content seems to be over 99.9% and is guaranteed 99.5%.

The price of recovered sulphur has virtually everywhere been adjusted to the new world price level of Frasch sulphur, especially as in most instances contracts are tied to United States or Mexican f.o.b. prices and a freight basis. The prices in France for sulphur recovered by the Société Nationale des Pétroles D'Aquitaine at about F. fr. 14,000 per metric ton, and in West Germany supplied by Ruhrgas A.G. and the oil companies of under D.M. 140 per metric ton are representative. Orkla Metal A./S. is reported to have made a further reduction of the price of pyrites-derived brimstone to Nor. Kr. 260 per metric ton f.o.b. Lökken, whilst in Sweden sulphur recovered from oil shales is on offer at Swed. Kr. 205-215 per metric ton. Canadian recovered sulphur is reported to be on offer at Can. $23.50 f.o.b. Vancouver.

The following table indicates broadly the delivered cost of brimstone of varying origin at principal consumers' destinations. The freight rates shown are either actual charters made at the end of December, or reliable indications.

f.o.b. Country of Origin	*U.K.*	*N.W. Europe*	*France (Mediterranean)*	*India*	*Australia*
U.S.A. $24—25 ...	(47s.) £11 —11⅛	(44s.) £10¼—11⅛	(48s.) £11 —11⅛	(85s.) £12¼—13⅛	(65s.) £11⅞—12⅛
Mexico $21.50—24	(47s.) £10⅛—11	(44s.) £ 9⅞—10¼	(48s.) £10⅛—11	(85s.) £11⅞—12¼	(65s.) £11 —11⅞
Italy Lire 23,000 per metric ton ...	(28s.) £14⅜	(25s.) £14⅝	(22s.) £14½	(55s.) £16⅛	
Canada $23.50 ...	(60s.) £11½	(55s.) £11¼	(55s.) £11¼	(70s.) £12	(60s.) £11½

Freight indications ( ) have been reduced to f.i.o. terms to provide a common basis for comparison.

Pyrites

The price level of European pyrites underwent a major reduction and the new prices range of 72s. 6d. - 82s. 6d. has taken effect as from the 1st January. The latest reduction is of the order 10/- per ton making the cumulative decline over the past 12 - 15 months 17/6 to 22/6 per ton equal to an average £2 per ton of sulphur.

Cyprus Mines Corporation pyrite concentrates, which in recent years have been in heavy demand, are now sold at 82/- per ton f.o.b. The lower residues values of Rio Tinto concentrates have caused their price to be dropped to 72/6, and those of Portuguese crude pyrites to 75/-, whilst the greater residues value of Spanish crude pyrites notably Tharsis and Rio Tinto is said to allow a quotation at 82/6 per metric ton f.o.b.

Persistently low freight rates coupled with advantageous residues values have substantially improved the overall status of pyrites in the eyes of consumers. Duisburger Kupferhütte, by successfully negotiating with Germany's iron makers a favourable price for her purple

ore, has assured Continental pyrites roasters a fair return which is enhanced by a voluntary reduction in treatment charges and as a result of which basic residues values appear to have declined only a few shillings to range from £4 to £4 10s. In the United Kingdom blast furnaces appear to have maintained, and in some instances even improved, the valuation of residues so that subject to transport cost up to 70 - per ton fines residues and 50/- flotation residues is realised. Low sea freights are allowing a large-scale movements of old residues stocks notably from Spain to United Kingdom and Germany.

In the United States, Canadian and domestic pyrites prices appear to have been adjusted to bring them into line with the new price level of brimstone.

Freights

Continued weakness characterised freight markets in recent months and lower coal shipments partly due to mild weather and the absence of significant grain or other commodity movement kept down the demand for shipping space. The United Kingdom Chamber of Shipping voyage index (1952=100) declined further from 81 in October to 71.6 at the end of December.

Despite the low freights, attractive to low cost commodities such as brimstone, pyrite, phosphate rock, etc., there was no increased activity as markets continue quiet if not weak. In recent months this applied particularly to pyrites owing to high consumer stocks and the expectations of lower prices in the New Year which proved to be justified. There are some signs that consecutive charters may be concluded at rates nearer current levels than was the case two or three months ago. Freights then fixed for early 1958 were based at 65s. for United States Gulf to United Kingdom; current spot rates to major U.K. ports are about 52s. and those to the Continent 44s. f.i.o.

Pyrites freights from Huelva to the Continent have declined to 23/6 and the differential for Eastern Mediterranean is only 4/6. Late eastward Atlantic charters were as low as $4.

Supplies

United States Frasch sulphur output was in recent months held at about 450,000 tons per month, but owing to lesser domestic and export sales, the level of production still exceeds disposals. In contrast, imports of Mexican brimstone are rising and sales of recovered brimstone are steadily expanding. The slowing down of industrial activity in the fourth quarter 1957 notably of steel, and rayon and pulp, adversely affects sulphuric acid demand and sulphur use in general, which in 1957 appears to have been about 8% lower than in the preceding year. The prospect of new Frasch sulphur capacity in the form of Texas Gulf Sulphur's Fannett dome starting in 1958 and Freeport Sulphur Company's two new major domes, Lake Pelto and Grand Isle, due in the next two years, is at the present time, to say the least, embarrassing to the United States industry.

In Mexico expansion of plant and the number of operating wells has raised output to a daily rate of about 3,700 tons. Vigorous export sales by Pan American Sulphur Company have temporarily brought stock building to a halt, while Gulf Sulphur Corporation has substantially improved the ratio of shipments to production. Rising domestic consumption is beginning to offer an outlet to the Frasch sulphur producers but the intention of Texas Gulf Sulphur Co. to start shipments from Nopalapa dome this year, and by Texas International Sulphur Co. to start production at Texistepec, coupled with the entry of Freeport Sulphur Company into the Mexican industry points to the continued rapid expansion of supplies and search for export outlets markets.

In Italy fused sulphur output — about 180,000 tons in 1957 — may further recede under pressure of low world market prices, although temporary relief by subsidy on production and exports is preventing an immediate crisis. Fused sulphur stocks, reduced to about 200,000 tons by the end of 1957, is almost commensurate with current sale commitments of Ente Zolfi Italiani. Expansion of sulphur ore production for acid manufacture, notably in Central Italy, is making slow but steady progress.

The Chilean industry is now virtually confined to the domestic market and production, is believed to have fallen below 30,000 tons while in the Argentine completion of a Chemical Construction Corporation ore refining

plant points to the early reduction of import needs by about 10,000 tons annually.

In Japan production after the steep rise early last year is now being cut back owing to severely curtailed demand. The rayon industry is especially hard hit by contracting world markets and has curtailed output of filament by 28% and of staple fibre by 25% so that her brimstone needs are at least 30,000 tons below last year's level.

Recovery

In Canada output of recovered brimstone from natural gas has risen to an annual rate of over 150,000 tons and is growing rapidly. Offers to consumers in British Columbia, not already using Alberta brimstone, and in North-West United States are accelerating and export offers to the Far East and Australasia are reported.

Greater oil refinery activity in the United Kingdom and Europe based on Middle East crude oils is reflected in increased sulphur recovery output. The oil refinery projects designed to meet the greater energy demand of the coming years all include sulphur recovery plant and more new capacity is due on stream progressively over the next three years. Sludge acid use is expanding, the latest addition being a Miley retort at Norddeutsche Affinerie, Hamburg. In the United States its use accounts for over one-half million tons per annum acid output and it is growing rapidly.

The decline of non-ferrous metal demand has resulted in a severe set-back of production throughout the world and in consequence by-product acid has been curtailed. Its present level is estimated about 30% below normal.

Pyrites

No significant increase in demand is likely in 1958. In Germany, the mainspring of expansion in recent years, distributed stocks are high and a pause in the steep rise in acid output is expected. Downward adjustment of stocks in the United Kingdom and in France is expected to affect at least the first six months deliveries. The principal European pyrites suppliers have ample capacity, but having lost some flexibility on account of narrower profit margins following the latest price cut, the majority will be hard hit if contracting markets entail an appreciable cut-back in output. An increase of pyrite production is reported from Rumania and Bulgaria. Turkish cupreous pyrites seek markets in Western Europe where Cuban ore already finds a ready outlet. Sulitjelma A.S., one of the leading Norwegian pyrites producers, is building a new 800 tons per day ore concentrating plant which will result in an increase of the company's output of flotation concentrate. In Canada the adverse trend of non-ferrous metals demand is reducing by-product pyrites output but supplies still far exceed demand. As the result of the 1,000 tons per day Noranda acid plant at the Blind River uranium field starting to consume pyrites in place of the temporary use of brimstone, the level of domestic pyrite consumption is rising and coupled with exports to United States and to Europe, an attractive market while freights are low, pyrite shipments from Canadian mines may approach three-quarters of a million short tons sulphur in 1958.

Summary

There is no indication as yet of a return to price stability although the threat to United States Frasch sulphur markets in the Mississippi valley appears to have been averted and to those on Atlantic Seaboard partly contained by the conciliatory gesture of the Mexican producers. The reduction of European pyrites prices and the maintenance of cinder values in the face of a sizeable drop in iron ore prices mainly on account of lower freights, has improved their competitive ability and appreciably slowed the pressure to convert to brimstone use.

World supplies of brimstone are rising more rapidly than demand and producer stocks at the beginning of 1957 are estimated at $5\frac{1}{2}$ million tons, an increase of about one-half million during 1957. Crude and by-product pyrites are in ample supply throughout the world and in Europe marketing under competitive conditions is orderly. With the rate of sulphur recovery expanding, the imminent completion of the major dome projects of the United States and the Mexican Frasch sulphur industries, underlines the weakness of the world sulphur market.

UNITED STATES SULPHUR

Frasch Sulphur Production

Production of Frasch sulphur in the third quarter of 1957 totalled 1,379,300 tons and, although 49,000 tons (3¾%) higher than in the previous quarter, it was 364,000 tons (21%) lower than in the corresponding period of last

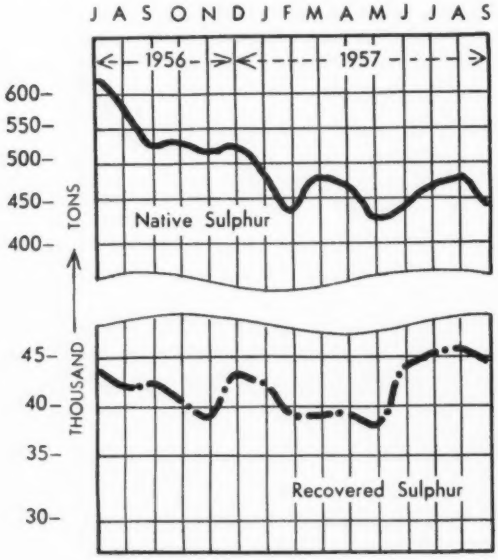


Fig. 3. Monthly production.

year, when the highest quarterly total on record was produced. The individual producers appear to have stabilised their aggregate output at a monthly average of about 450,000 tons, and Frasch sulphur production during 1957 is expected to total 5½ million tons.

Texas Gulf Sulphur Company is reported to have adjusted operating rates at Boling Dome, Spindletop and Moss Bluff so that, including her share of the output at Long Point Dome, the Company's production in 1957 will total 3 million tons. New facilities are being installed at Spindletop for the shipment of liquid sulphur in barges and larger vessels. The new Frasch sulphur plant at Fannett Dome, Jefferson County, Texas, has been completed. Designed for a water capacity of 2 million gallons daily, it is reported to contain several features designed to improve operating efficiency. The plant has been tested and production is expected to start early in 1958.

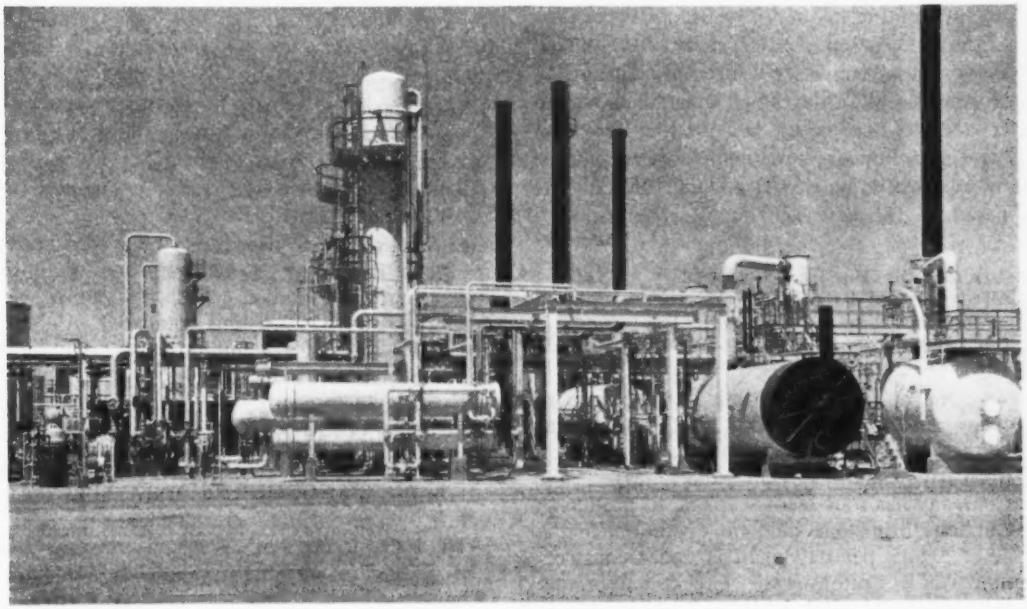
Sales and Consumption

Apparent sales of Frasch sulphur of 1,193,356 tons represent one of the lowest quarterly sales totals in recent years, and sales were 200,000 tons (14¼%) lower compared with the corresponding period of last year. The decline is attributable in almost equal parts to lower brimstone exports and to lesser shipments to domestic users. Domestic consumption in the third quarter appears to have contracted only a little and the difference may be attributed principally to increased imports of Mexican brimstone—about 55,000 tons higher than a year ago—and to a lesser extent to the pressure of recovered sulphur supplies. The index of United States industrial activity of 144 (1947/9=100) in the third quarter was slightly higher than last year, but there were several weak spots in the economy which included prominent sulphur consumers such as the rayon, steel and the pulp and paper industries. Non-acid uses appear to have declined appreciably, whilst sulphur use for sulphuric acid manufacture was unusually high for the summer quarter.

Sulphuric Acid

Compared with 3.58 million short tons (gross 100% H_2SO_4) in the corresponding period last year sulphuric acid production totalled 3.94 million short tons, an increase of 10%. The lower level of acid demand from the rayon and steel industries was, at the beginning of the quarter more than offset by the increased acid use in fertiliser manufacture. Production of ammonium sulphate was at about the same level as last year, while superphosphate production following the depletion of stocks in the late Spring and in preparation for the 1957/8 fertiliser season reached a higher level than customary in the third quarter. The preponderance of single superphosphate supplies has recently fallen, and in July concentrated superphosphates for the first time exceeded the level of single superphosphates. This development is not as pronounced in the Western States, partly on account of the more widespread occurrence of alkaline soils which need the greater quantity of sulphur present in single superphosphate.

To meet the demand for granular and pelletized fertilisers, Stauffer Chemical Company has added new equipment at their Richmond, California, plant, which now has an annual production of 50,000 tons pelletized or granular single superphosphate. There has been a steady demand for sulphuric acid from the oil refining, uranium and titanium pigments producers, and in the latter half of the quarter several acid makers had some difficulty in meeting their commitments. Throughout the quarter, rising costs threatened an increase in acid prices, but the reduction of domestic brimstone price on 18th September arrested this trend. Apart from tied contracts, notably in the fertiliser industry, acid producers have shown no intention so far of lowering their prices following reduction of brimstone prices as they claim that profit margins remain too slender to permit it.



Hancock Chemical Co. General view of chemical plant at Watson, California, which now has a daily capacity of 150 tons brimstone.

Exports

Brimstone exports of 359,768 tons in the third quarter were 103,000 tons less than the corresponding period last year. This drop appears to be the first telling impact of the world-wide competition on United States exports which for the past three years have remained practically constant at the high annual rate of about 1.6 million tons. During the nine months of this year exports totalled 1,107,232 tons compared with 1,215,895 tons shipped from January to September 1956. Of their most important markets, United States exporters failed to maintain their position in the United Kingdom, France, Switzerland, Australia and New Zealand. In the expanding markets, principally Belgium, Holland, West Germany and South Africa, sales have increased, but only after Mexican exports and the growing numbers of indigenous producers of recovered sulphur had claimed a share of the market.

Recovered Sulphur

Production of recovered sulphur in the third quarter of 1957 reached a record total of 135,928 tons, 9¼% greater than the previous quarter and 8,000 tons greater than the corresponding period last year. This steadily

expanding sector of the industry is expected to contribute in 1957 about 530,000 tons high purity brimstone to the domestic supplies in addition to a substantial volume of sulphur in H_2S for direct use in acid manufacture. The rising level of activity at the Tidewater Oil Company's refinery at Delaware, which incorporates the world's largest sulphur recovery plant, results in an increasing tonnage of sulphur becoming available in the biggest local acid sulphur market in the U.S.A. Similarly increased contributions to recovered sulphur supplies originate from the following plants which, in 1957, have increased their capacities: Hancock Chemical Company at Watson, California, now has a daily capacity of 150 tons brimstone; Gulf Oil Corporation at the Port Arthur, Texas, refinery has doubled capacity to 120 tons; Wilshire Oil Company at Norwalk, California, has increased the plant capacity to 20 tons per day.

The following new plants are scheduled to start operating at the end of 1957, all are based on H_2S extracted at oil refineries:—

		Brimstone tons per day
American Oil Company	Yorktown, Va.	60
Allied Chemical & Dye Corporation	Bayway N.J.	50
Anlin Company ...	Perth Amboy N.J.	35
Pontiac Eastern ...	Hattiesburg, Miss.	25

One of the most prominent producers of recovered brimstone from sour natural gas is the Pan American Petroleum Corporation (formerly Stanolind Oil and Gas). The Company has four plants capable of producing 150 tons brimstone daily in operation, and a new 25 tons per day plant is under construction at Cottonwood Creek, Worland, Wyoming, and is due to come on stream in March 1958.

Apparent sales of recovered sulphur in the third quarter totalled 127,741 tons and were 5,655 tons (4½%) greater than the corresponding period last year. Stocks continued to increase and have now risen to the record level of 152,940 tons. Some 35,000 tons (29%) greater than at the end of the third quarter 1956, the increase is thought to be primarily attributable to the greater number of operating plants and to the need to safeguard sales commitments against the customary fluctuations in refinery operations. Recovered sulphur stocks still only represent 17 weeks deliveries compared with 43 weeks in the case of Frasch sulphur.

Summary

(a) The level of Frasch sulphur operations having been reduced, production of native and recovered brimstone in the third quarter totalled 1,515,228 tons.

(b) Imports of Mexican Frasch sulphur have risen to a quarterly rate of 110,000 tons; twice as much as last year.

(c) Overall brimstone consumption did not decline appreciably in the third quarter—the drop in non-acid sulphur demand being almost balanced by greater acid sulphur usage—but apparent sales of domestic Frasch and recovered sulphur totalled only 1,321,097 tons, nearly 200,000 (14½%) less than in the third quarter 1956.

(d) Export of sulphur at 359,768 tons were 103,000 tons (22%) lower than the third quarter 1956, mainly on account of Mexican competition, but also due to greater indigenous recovered sulphur supplies in importing countries.

(e) Stocks of U.S. native and recovered brimstone continued to increase, and on 30th September, totalled 4,425,498 tons, an increase of 627,000 tons compared with one year ago, and 1.3 million tons more than in September 1955.

REVIEW OF 1957

***By* Mr. LANGBOURNE M. WILLIAMS**

IN HIS traditional year-end review, the President of Freeport Sulphur Company commented as follows:—

"The demand in the United States for sulphur, a traditional barometer of industrial activity, declined slightly in 1957 but remained near the record high established the previous year. Exports of U.S. sulphur also were high. Consumption of sulphur in all forms in the United States was down about 5% from the record 5,780,000 tons consumed in 1956. Exports from the U.S. are estimated to be within about 100,000 tons of the 1956 high of 1,650,000 tons.

"The slight decline in the use of sulphur reflected the levelling off of business experienced

by major consuming industries such as steel, fertilizer, chemicals, paper, pigments, and rayon. As in the past years, approximately 80% of the sulphur consumed was converted into sulphuric acid and the balance was used in elemental form or in other compounds. The bulk of the sulphur consumed by the nation was mined by the Frasch hot water process from deposits in Louisiana and Texas with some being imported from Mexico and the remainder being sulphur obtained in various forms from other sources. Total output of sulphur from all U.S. sources amounted to an estimated 6,900,000 tons, compared with 7,820,000 tons in 1956. Frasch sulphur alone accounted for 5,500,000 tons, down from the 1956 high of 6,425,000 tons. Of the remainder of the supply 525,000 tons represented elemental sulphur recovered from gases, 425,000 tons sulphur contained in pyrites, and 450,000 tons sulphur in other forms.

"Eleven U.S. Frasch process mines were in operation throughout the year, and a twelfth, the Damon property of Standard Sulphur Company in Texas, operated until April, when it was shut down. Texas Gulf Sulphur Company remained the largest producer, followed by Freeport, Jefferson Lake Sulphur Company, the Duval Sulphur and Potash Company.

"Three new U.S. deposits are under development. The largest is Freeport's Grand Isle, a discovery of major magnitude located off the Louisiana coast in 45 feet of water six miles from the nearest land. Grand Isle will be the first completely off-shore sulphur mine and will incorporate many unique features designed to make it one of the industry's most efficient operations.

"In September Texas Gulf announced a sulphur price reduction to meet general competitive conditions. Mexican producers had been offering sulphur at substantially under the U.S. scale. The Texas Gulf announcement was followed by similar action by other U.S. producers. The price reduction amounted to $3 per ton, or somewhat over 10 per cent."

FREEPORT SULPHUR COMPANY

NEW DOME DEVELOPMENTS

AT present, Freeport Sulphur Company operates four domes, of which two, Grande Ecaille and Garden Island Bay, are among the largest and most efficient in the industry and account for the bulk of the company's annual output of over 2 million tons. The smaller mines are Bay Ste. Elaine and Chacahoula.

Two new domes are now under development and promise to be important on account of their inferred size, whilst their unusual construction and engineering aspects represent further pioneering achievements by this, the oldest active Frasch sulphur producing company.

Lake Pelto

The Lake Pelto property lies under six to eight feet of water and is separated from the open waters of the Gulf of Mexico by only a narrow strip of sand. It is the first completely submerged deposit to be exploited. Wells have been drilled from floating rigs and equipped for future production, and a small island has been filled and prepared for the installation of the mining plant and ancillary facilities. Upon completion of the mining of the Bay Ste. Elaine deposit, the barge-mounted plant in use at that dome will be moved to Lake Pelto.

Its location has put it beyond the reach of an economic supply of large quantities of fresh water, until recently a necessity for the Frasch process. The Bay Ste. Elaine plant, however, already incorporates a process conceived and perfected by the company to utilise sea water, whereby this is heated to high temperatures without the usual detrimental effects of corrosion.

Another new aspect of this dome development is the drilling of directional wells. Directional wells are more costly to drill than conventional vertical wells, but since it is

possible to sink and equip a considerable number from a single location over-all costs are actually reduced. This innovation reduces to a minimum the moving of drilling rigs, it reduces the lengths of walkways and piping to connect each well-head and it makes it possible to reach ore bodies which might otherwise be inaccessible.

As Lake Pelto lies closer to the open Gulf than any other sulphur mine, special precautions are being taken to guard it from

Drilling rig at Lake Pelto dome.

the high waters and winds which are prevalent in the area during the annual hurricane season.

Since there will be no adequate land area on which to store the sulphur at the mine the output will be handled in the same manner as that of Bay Ste. Elaine and Garden Island Bay. As it comes from the wells, molten sulphur will be pumped into 1,000-ton insulated barges which will be towed to Port Sulphur, some 70 miles away, where the liquid will be sprayed into storage vats to cool and solidify.

Grand Isle

Grand Isle represents a notable step forward, even compared with such technologically-advanced mines as Bay Ste. Elaine and Lake Pelto. It will be the first offshore sulphur mine in history and it is also intended to be one of the world's most efficient and low cost sulphur operations.

Actual construction of the large man-made steel island is scheduled to begin early in 1958. When completed, the Y-shaped structure will stretch almost a mile in total length. Steel towers formed by piling sunk into the floor of the Gulf beneath the 45-feet deep water will support bridges for walkways and platforms 55 feet above the normal Gulf level, well out of reach from hurricane waves. Mounted on the island will be the mining plant designed to produce several million gallons of superheated water a day, plus compressed air and electric power, accommodation for sleeping and feeding employees, a helicopter landing platform, drilling rigs and accompanying pipe storage space, sulphur collecting and temporary storage tanks, and many miles of piping to carry air, water and sulphur.

The mining plant will use Freeport's process for heating sea water using as fuel natural gas supplied by means of a submerged pipe leading out from the mainland.

All the sulphur wells at Grand Isle will be directionally drilled. Initially they will be sunk from two platforms located on the ends of the arms of the Y-shaped structure. From these platforms wells will reach out in an area sufficiently large to tap reserves required for several years. Later a third platform is contemplated to reach additional reserves. The problem of subsidence, which is faced at every sulphur mine, will be handled by a novel method of jacking-up and lengthening the support piling. Employees will work on five-days-on, five-days-off shifts. They will be carried to and from the island by company helicopters. The output will be delivered to storage at Port Sulphur, 33 miles away, by insulated barges half as large again as those now in use by the company.

SULPHUR IN MEXICO

THE vigorous development of the Mexican Sulphur Industry was maintained during the autumn months. On the basis of plans initiated earlier in the year the three active Frasch sulphur producers—Pan American Sulphur Company, Gulf Sulphur Corporation and Texas Gulf Sulphur Company—strengthened their production facilities, and daily Frasch sulphur output frequently exceeded 3,700 tons, equal to an annual rate in excess of 1.3 million tons.

During the third quarter, sulphur production in Mexico totalled about 245,000 tons; of this 236,881 tons was represented by Frasch sulphur, about 7,000 tons was recovered by PEMEX from oil and natural gas, and about 1,000 tons was derived from native sulphur ores in Central and Western Mexico. Frasch sulphur output was 8,000 tons (3½%) greater than in the preceding quarter and 12,000 tons (5½%) greater than in the third quarter of 1956. In November, total sulphur output exceeded 100,000 tons.

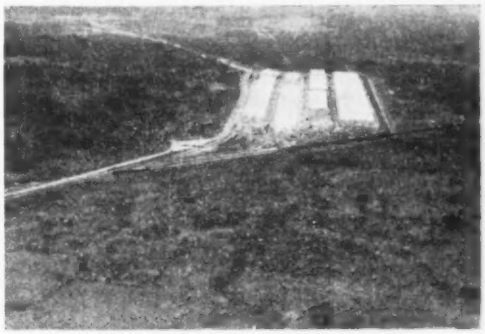
Pan American Sulphur Company provides about 70% of the Frasch sulphur output in Mexico and is firmly established as the third largest brimstone producer in the world. The company's Jaltipan dome, together with the Las Salinas dome of Gulf Sulphur Corporation, are at present the sole suppliers of Mexican Frasch sulphur as the output from Nopalapa dome continues to be stockpiled by Texas Gulf Sulphur Company. The entry of a fourth active producer, Texas International Sulphur Company, is anticipated in the course of 1958. Following the successful conclusion of long drawn-out negotiations between Sulphur Exploration Company — a major concession holder — and Freeport Sulphur Company, the latter may now be expected to start a vigorous development programme with the object of securing a low cost dome on the sulphur-rich Isthmus of Tehuantepec.

Domestic sulphur consumption in Mexico is being stimulated by the rising level of industrial activity, and a fair amount of investment capital, some of it foreign, is entering the various sulphur-using industries accelerating their expansion and diversification. Current consumption is at an annual rate of 77,000 tons, and is expected to rise to an annual rate of 150,000 tons during the first half of 1958, and possibly to 200,000 tons by early 1959.

PAN AMERICAN SULPHUR COMPANY

The leading position of this company in Mexico's young sulphur industry is highlighted in the activity report for the third quarter of 1957, presented by the President, Mr. Harry C. Webb.

Production totalled about 168,000 tons, bringing output for the nine months of 1957 to 515,389 tons, an increase of 83,670 tons (19%) compared with the same period in 1956. Shipments of 496,000 tons were 185,000 tons (60%) greater than in the first nine months of 1956. In the third quarter 187,000 tons was shipped, aided by the record tonnage of 72,000 achieved in September, which represents the greatest single monthly movement of sulphur since the company started operating. About 43% of the company's deliveries are directed to consumers in the United States, where her sales prospects are prompted by the success of deliveries from the stockpile at Tampa and from another now established along the Eastern seaboard. The excess of shipments over production caused a slight drop in the company's mine and port stocks, which at the end of September totalled 512,000 tons. The company's part in expanding production and shipping facilities is impressive. The number of wells equipped for production has been increased by one-half and they now total 15 and Relay Station No. 2, built one year ago, was put into operation. These developments



Pan American Sulphur Co. storage vats.

taken in conjunction with the increased water facilities enables the company to raise production at will to 3,000 tons daily. The completion of the second loading installation at Coatzacoalcos foreseen for November was reported to have been realised on time, and the company is now able to load and ship over one million tons annually. Greater sales revenue—$542,000 up in the third quarter—was almost balanced by greater costs and provision for taxes. The first nine months' revenue of $11.8 million—$4.35 million more than in 1956—resulted in a net income of $2.56 million, equal to $1.27 per share compared with $0.81 per share earned in the first nine months of 1956. Mr. Harry C. Webb, expressing satisfaction with results achieved, looked forward to the year's output reaching 700,000 tons; preliminary results point to sales having reached a similar level.

GULF SULPHUR CORPORATION

At the company's Las Salinas dome the exploitation of new wells and the effect of the additional pumping station became apparent in the October and November results, when output totalled 21,000 and 20,000 tons respectively. The average daily rate of about 700 tons was frequently exceeded, and the design rate of 1,000 tons is reported to have been exceeded for the first time on 4th November when output reached 1,023 tons.

In the course of exploiting the new wells, none of the difficulties of excessive well pressure which afflicted earlier operations were experienced. Seven out of ten wells are reported to have yielded crude sulphur containing no more than 0.3% carbon and 0.05% ash, and it is expected that if filtered the material may compare favourably with brimstone from Texas Gulf Sulphur Company's Moss Bluff dome. In the meantime, it is understood that it will be set aside in a vat to meet special customer requirements.

The water transport facilities, which were recently strengthened by the provision of a new tug, are reported to be adequate to move a daily output of 1,000 tons which, in the liquid state, is carried to the company's stockpile at Coatzacoalcos. Export shipments in the third quarter totalled nearly 41,000 tons, of which two-thirds was directed to the United States and one-third to United Kingdom and Israel.

TEXAS GULF SULPHUR COMPANY

Results of the first eight months' exploitation of the Nopalapa dome are considered satisfactory by the company. The water heating plant of new design, mounted on twin barges, has come up to all expectations. At present the plant is producing at an annual rate of 170,000 tons brimstone and the entire output is being accumulated in a vat near the mine site. Cia Exploradora del Istmo S.A., the operating company and affiliate of Texas Gulf Sulphur Company expect to start shipment sometime in 1958.

FREEPORT SULPHUR COMPANY

The agreement signed between The Sulphur Exploration Company of Houston and the Freeport Sulphur Company at the end of September gives the latter an opportunity to develop resources in this new and promising production zone. The Sulphur Exploration Company holds large concessions on the customary terms from the Mexican Government whereby a portion of the concession—in this case one-tenth—reverts to the Government each year and the remainder must be intensively surveyed. The agreement provides for an extensive drilling programme by Freeport during 1958 to ensure that sulphur-bearing concession areas are not abandoned to the Government.

According to Mr. Kaplan, President of The Sulphur Exploration Company, Freeport has option to drill at least 26 wells during 1958 in the concession areas, and he expected that work would be concentrated on the South-East part where previous drilling had disclosed the existence of major sulphur formations. Under the agreement which, like The Sulphur Exploration Company's concession, is believed to be valid initially for five years, Freeport Sulphur Company, in the event of sulphur being exploited in commercial quantities, will share profits with The Sulphur Exploration Company.

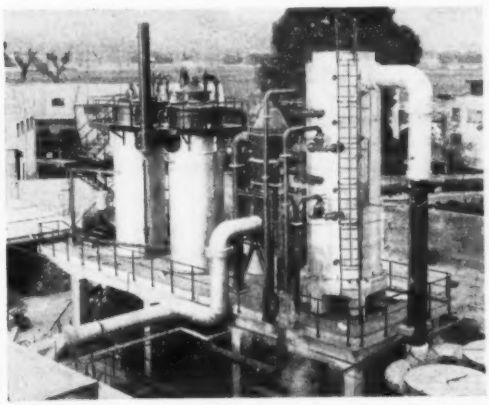
Domestic Consumption

The sulphur using industries of Mexico are making rapid strides and already their achievements are noteworthy. The fertiliser sector increased its output from 56,000 tons in 1950 to 212,000 in 1956, consisting of 72,500 tons superphosphates, 80,000 sulphate of ammonia and 60,000 tons of compounds. This output

level said to represent 62% of domestic fertiliser use of 34.1kgs. per hectare of cultivated land, leaves some 133,000 tons to be imported. Five new plants of which one is to produce triple superphosphate, one a mixed fertiliser and three nitrogen fertilisers are designed to add over 200,000 tons to Mexican supplies which will obviate the need to import and possibly after 1959 provide a modest export surplus.

	Capacity 100% H_2SO_4	*Production metric tons*	*% of utilisation*
1950	63,145	43,374	69
1951	128,845	56,667	44
1952	136,145	92,208	68
1953	119,650	102,503	86
1954	126,850	109,962	87
1955	161,200	124,887	77
1956	194,040	161,483	83

Sulphuric acid output of 161,500 metric tons in 1956 originated at 13 works which operated at 83% of installed capacity. In addition to new plants to meet the sulphuric acid needs of some of the above fertiliser plants, construction of several other sulphuric acid plants is foreseen before 1960. These include installations to meet the needs of the man-made fibre industries. Current sulphur needs of Mexico are estimated to total about 70,000 tons per annum, about two-fifths of which is derived from the sulphur recovery plants of PEMEX at Poza Rica. Consumption is expected to rise, slowly at first, to about one-quarter million tons in 1960.



Alkamex S.A. The 20 tons per day acid plant recently completed by Chemiebau Dr. Zieren at Mexico City.

Titanium Dioxide

The rising level of titanium pigments consumption and in particular in the period 1954-1956 steeply to 4,386 metric tons, prompted Du Pont S.A. de C.V., a subsidiary of E.I. Du Pont de Nemours of Delaware, U.S.A., to initiate plans in March, 1957, for the construction of a $2.8 million plant designed to produce initially 5,000 tons pigment and capable of being extended to 7,000 tons. In the early autumn, Industrias Quimicas Basicas S.A., announced a similar project costing $6.5 million and with an annual capacity of 8,000 tons titanium pigment in respect of which the Glidden Company of Baltimore is reported to have concluded a 10-year agreement to provide technical know-how. Both projects have incorporated sulphuric acid plants, with a capacity of up to 100 tons per day.

Independent observers contend that the Mexican market is as yet too narrow to accommodate both plants, which are said to be planned for construction on sites in the State of Vera Cruz, probably near Coatzacoalcos; neither has as yet been started, but it is believed that the first plant on-stream may get Government protection, although Industrias Quimicas Basicas S.A. may have additional consideration since one third of the company's share capital is said to be State owned.

Exports

During the first nine months of 1957 brimstone exports from Mexico totalled 631,707 metric tons, of which 232,974 tons was shipped in the third quarter.* Over one-half (53%) was shipped to United States, and of the remainder the major quantities were directed to Australia and New Zealand (12%), United Kingdom (11%), France (9%) and Holland (5%). Germany and Israel were new customers in 1957 and most established users increased their purchases, while deliveries to South Africa were reduced.

It is believed that Mexico's export volume in 1957 amounted to a little under 900,000 tons. The supply of nearly one-half million tons Mexican Frasch sulphur to United States consumers is having serious repercussion on the order books of United States producers, and now that Mexico's overseas order book has risen to 400,000 tons United States sulphur exporters are adversely affected in these markets as well. Continued expansions point to pressure for new markets which caus

* Excluding sulphur in anhydrite consumed in the direct manufacture of sulphate of ammonia.

UNITED KINGDOM SULPHUR

Sulphur supplies

Brimstone: Imports of elemental sulphur during the third quarter 1957 amounted to 95,284 tons, and although 3,500 tons lower than during the corresponding period of 1956, they were 18,500 tons (24%) greater than in the preceding quarter. The higher level of imports was partly due to more favourable chartering conditions earlier in the year, when sea freights started declining. The additional imported supplies were, moreover, needed to compensate for the reduction of deliveries from the Government stockpiles which are now nearly run-down. With the lowering of sea freights first making itself felt during the third quarter the unit value of brimstone imports receded to £14 6s. 0d. per ton, an average of £1 14s. 0d. less than in the preceding quarter. None of the shipments which reached this country in the third quarter were affected by the price reductions towards the end of September. Imports of Mexican sulphur at 35,753 tons were valued at £13 14s. 6d., while those from the United States, totalling 59,989 tons, averaged £14 13s. 0d. Imports of nearly 4,000 tons filtercake, supplied by the Freeport Sulphur Company, augmented domestic waste supplies which Nitrogen Fertilisers Ltd., Flixborough, are able to use effectively in their specially designed acid plant.

Domestic recovered sulphur supplies regained if not exceeded their former level which had fallen as a result of greater use of Carribean crudes after Suez, and, in the absence of official statistics, it is estimated that in the third quarter production totalled 14,000-15,000 tons. Deliveries of refinery gas from Esso oil refinery, Fawley, to the Southern Gas Board, and from Shellhaven to North Thames Gas Board will result in additional brimstone and spent oxide being produced in the course of purifying these supplies to town gas standards.

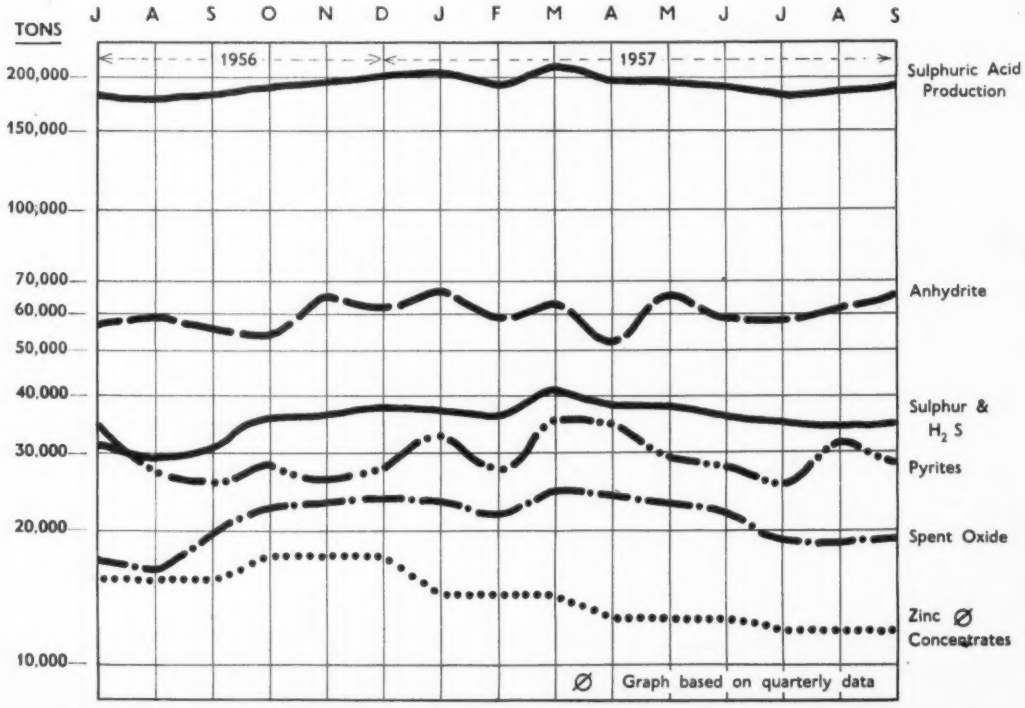


Fig. 4.

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Pyrites: Imports during the third quarter totalled 96,776 tons, 4% less than in the second quarter, to raise the first nine months' pyrite imports to 246,000 tons, 14% less than in the same period of 1956. Under the impact of lower sea freight and, in the case of Spanish pyrites, the lower f.o.b. prices which were reduced by 7/6d. per ton as from 1st July, 1957, the average unit value of imports in the third quarter was £6 10s. per ton compared with the peak of £8 0s. in the second quarter. Imports of Cypriot pyrites accounted for over one-half of the total, and in addition to Canadian and Spanish pyrites, some 4,300 tons of Bolidens pyrites entered the United Kingdom.

Industrial production advanced during the summer months and the seasonally adjusted index—3-4 index points higher than in corresponding months of 1956—rose to 140 in September. Imported supplies met about 58% of sulphur needs—38½% in the form of brimstone, 18½% as pyrites, and 1% as filter cake—and indigenous resources, including sulphur in anhydrite, spent oxide, and zinc smelter gases and sulphur recovered from coal and oil refining gases accounted for the balance of 42%.

Consumption of various raw materials during the third quarter compared with the relevant earlier periods are given below:—

	3rd Qtr. 1957	*2nd Qtr. 1957*	*% Change*	*3rd Qtr. 1956*	*% Change*
Elemental Sulphur					
Acid: Imported	65,900	75,400	−12½	58,200	+13¼
Recovered H_2S & Filter Cake	11,400	9,100	+25¼	7,500	+52
Total Acid Sulphur	77,300	84,500	− 8½	65,700	+17½
Regular	27,000	29,000	− 7	26,100	+ 3½
Total Elemental Sulphur	104,300	113,500	− 7	91,800	+13½
Pyrites	85,900	92,200	− 6¼	87,900	— 2¼
Spent Oxide	58,900	68,400	−14	56,100	+ 5
Anhydrite	183,600	176,300	+ 4¼	172,000	+ 6¾
Zinc Concentrates	36,100	37,800	− 4½	45,000	−19¾

Zinc Concentrates: Zinc smelter operations further declined in the third quarter on account of the weakness of the metal markets, and in consequence by-product acid manufacture receded to about 28,000 tons (100% H_2SO_4), about 70% of capacity, and the lowest level since 1951.

Stocks: Acid sulphur stocks rose about 5,000 tons during the third quarter and together with non-acid sulphur stocks estimated at 15,000 tons, brimstone stocks totalled about 110,000 tons or a little over 13 weeks' supply at prevailing rates of usage. Pyrites stocks rose 15,600 tons (7½%) to 229,100 tons or 35 weeks' supply, whilst spent oxide stocks designated for acid manufacture continued to decline—1,400 tons in three months—to 114,900 tons or 25 weeks' supply.

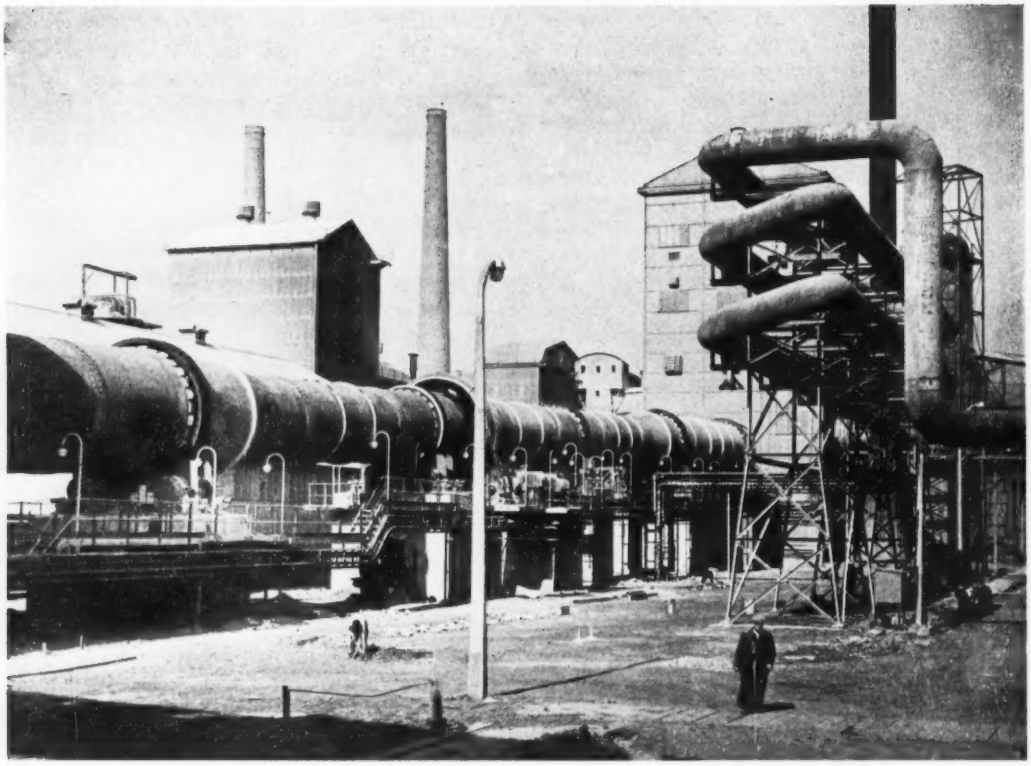
Consumption

During the third quarter 1957 consumption of sulphur in all forms* totalled about 223,000 tons, an increase of 12,000 tons (5½%) compared with the corresponding period of 1956. Brimstone consumption—including H_2S and filter cake—totalled 104,300 tons, the highest tonnage on record for the third quarter and 13½% greater than in 1956. Pyrites consumption at an annual rate of about 340,000 tons, was the lowest for any quarter since the beginning of 1953, mainly on account of high acid sulphur use. Anhydrite consumption for acid manufacture of 183,600 tons was the second highest on record, and spent oxide, whilst seasonally lower, also showed an advance (5%) over consumption in the same quarter of 1956.

Regular Sulphur

Reduced demand by the rayon industry continued to affect adversely the level of non-acid sulphur consumption. Much of the slight rise in output over last year's is accounted for by synthetic fibres and unused rayon capacity is now estimated at 100 million lbs. or about one-fifth of installed capacity. Deliveries of filament yarn were 10% lower in the third quarter and, but for the motor industry boom which caused an 8%

* Excluding sulphur in anhydrite consumed in the direct manufacture of sulphate of ammonia.



Imperial Chemical Industries Ltd. Anhydrite acid plant at Billingham. The new 350ft. kiln with an annual consumption of 130,000 tons anhydrite.

increase for high tenacity yarn, these might have receded as much as staple fibre, deliveries of which were 25% below those of the first quarter. Requirements for rubber compounding sulphur dioxide and sulphites are reported to have been above average.

Sulphuric Acid*

Production of sulphuric acid in the third quarter totalled 545,828 tons (100% H_2SO_4), 5¾% more than in the corresponding period of 1956. It resulted from the use of the following raw materials :—

	3rd Qtr. 1957 %	*2nd Qtr. 1957* %	*3rd Qtr. 1956* %
Elemental Sulphur			
Imported brimstone ...	33¼	36	31¼
Recovered, H_2S and filter cake	5¼	4¼	4
Sulphur in:—			
Pyrites	20¼	21	22¾
Spent oxide	14½	15½	14½
Anhydrite	19¾	17¾	19¾
Zinc concentrates	5½	5½	7¼

The seasonally intensive employment of anhydrite acid capacity resulted in an increase of its share of the total production to 19¾%. Compared with the previous summer quarter the share of brimstone acid output has also increased from 35¾% to 39½%, at the expense of pyrites acid output and by-product acid at zinc smelters. There was no significant change in the effective production capacity of 698,040 tons (100% H_2SO_4); only 78.2% was employed. More chamber plants were idle and employment of 63.6% compares with 83.2% of contact acid capacity.

Sulphuric Acid Consumption

Consumption of sulphuric acid in the third quarter amounted to 553,738 tons (100% H_2SO_4), an increase of 8% over 1956 and the highest third quarter's total on record.

Principal changes in individual consuming industries compared with the corresponding period in 1956 were as follows :—

* We are indebted to the National Sulphuric Acid Association for information placed at our disposal

	Change tons	Change %
Titanium dioxide	+ 12,200	18
Soap, Glycerine and Detergents	+ 8,400	46½
Superphosphates	+ 8,200	7¾
Sulphate of Ammonia	+ 5,400	7¾
Dyestuffs and Intermediates	+ 3,400	19½
Iron pickling	+ 2,600	9½
Rayon and Transparent paper	+ 2,600	4
Tar and Benzole	+ 2,000	34

Quantitatively smaller, but nevertheless important changes occurred in bichromates explosives (−21½%), hydrofluoric acid (−32%).

Acid usage by the main consumer groups in the two periods was as follows :—

	3rd Qtr. 1957 000 tons	%	3rd Qtr. 1956 000 tons	%
Fertilisers	188	34.0	175	34.1
Fertilisers				
Superphosphates	113	20.4	105	20.5
Sulphate of Ammonia	75	13.6	70	13.6
Chemicals	61	11.0	69	13.5
Titanium dioxide and Lithopone	82	14.9	71	13.8
Rayon and Transparent Paper	66	11.9	63	12.4
Metallurgy	31	5.7	29	5.7
Oil Refining	15	2.7	15	3.0
Dyes	21	3.8	18	3.4
Soaps & Detergents	26	4.8	18	3.4
Miscellaneous	64	12.2	54	10.6
Total	554	100.0	512	100.0

As the result of the progressive expansion of sulphuric acid consumption for titanium dioxide manufacture, the volume of acid consumed is at present only exceeded by that used in superphosphates manufacture. The largest proportionate rise in acid use can be recorded in the soap and detergent industry; developments in detergent intermediates manufacture are believed to account for the spectacular growth in this use sector. Sulphuric acid demand in iron and steel manufacture has been limited by regeneration of acid pickle liquor, whilst new processes in which sulphuric acid plays a lesser part are believed to have influenced explosive output.

Summary

Significant developments in the United Kingdom industry in the third quarter 1957 are as follows :—

(a) Consumption of sulphur in all forms of 223,000 tons was the highest in any summer quarter.

(b) Import prices of brimstone and pyrite are lower following the drop in sea freights.

(c) Sulphuric acid output has expanded while non-acid sulphur use, on account of the difficulties of the viscose rayon industry, is stagnating.

(d) The continued high level of brimstone and anhydrite acid production contrasts with the recession in pyrites acid and by-product acid manufacture, the latter caused by reduced metal demand.

The anticipated trend in the consumption pattern of the United Kingdom sulphur industry, namely the full use of available anhydrite acid facilities and the progressive growth of brimstone use, is now firmly established. Of the acid plant projects initiated since the cessation of Government control on 31st December 1955 — new brimstone acid installations have been placed on stream by Messrs. R. & J. Garroway at Glasgow and British Titan Products Co. Ltd. at Grimsby. At least three additional similar plants with a combined capacity of 425 tons per day are under construction and due to start operations during this year. At the same time the prospects of pyrites usage have improved to the extent that at established modern plants, favourably situated to blast furnaces or other equitable cinder outlets, there is no longer a sufficiently clear-cut cost differential to make conversion to brimstone use attractive. The further reduction of pyrites prices in the New Year helps this position and, aided by better residue valuation, the possibility of pyrites being selectively competitive with brimstone is in sight.

Adequate acid plant capacity and cheaper raw materials have promoted an ample supply situation as the result of which the United Kingdom sulphuric acid market has weakened, especially in the North of England, during the autumn months. Greater labour and overhead costs, however, have so far prevented a break of the higher market price, established last August. The increasing competition in world sulphur markets favours the United Kingdom industry and as the world's largest importer — next to the United States — it is at present the focus of attention by active and potential suppliers of brimstone and, to a lesser extent, pyrites. The concurrent expansion of brimstone and H_2S recovery facilities from oil and coal gases as a result of which output in this sector of indigenous sulphur supplies is likely to double speedily, is accentuating the buyers' market.

W. & H. M. GOULDING LIMITED

One hundred years of fertiliser production in Ireland

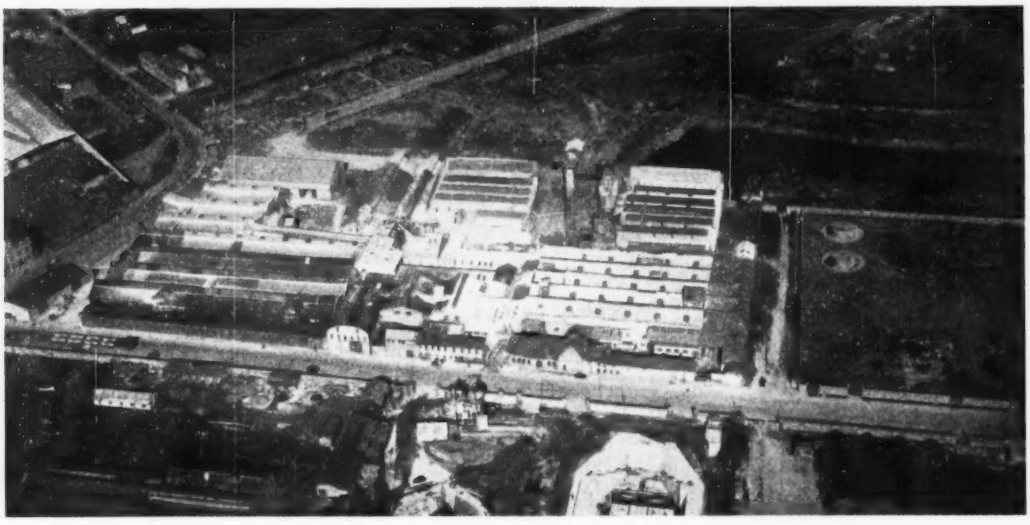
ONE of the first companies to produce "chemical manures" on a commercial scale, W. & H. M. Goulding Ltd., of Dublin, Ireland, celebrated their centenary last year.

Two brothers, William and Humphrey Manders Goulding, produced small quantities of superphosphate in the early 1850's as a sideline to their business as Oil and Colour Merchants and agents for agricultural materials. The success of this venture led in 1856 to the acquisition of premises at The Glen, Cork, where manufacture of superphosphate was commenced from bones obtained locally and imported sulphuric acid. This importation of acid was a serious handicap, and in 1860 an acid plant burning sulphur and consisting of one lead chamber was installed. The sulphur was soon replaced by the cheaper pyrites obtainable from Avoca, Co. Wicklow, and by 1868 the plant had expanded to five chambers. This expansion was reflected in sales of fertiliser, which rose from 857 tons in 1861 to over 7,000 tons in 1868.

By 1869, a second factory, complete with acid plant, had been established in Dublin and within three years sales had more than doubled.

Phosphate rock gradually replaced ground bones and was obtained from a wide variety of sources. Rock from the United States was used extensively before supplies from North Africa were obtained regularly in the 1920's. Believed to be the first company to export fertiliser to the United States, extensive trade was also built up in the United Kingdom, France, Portugal, Russia, Norway and Natal, where a special fertiliser was sold for the sugar cane crop. The large trade with America led to a factory being opened at Pensacola, Florida, in 1891 with a capacity of 45,000 tons. This was operated until 1905 when, for various reasons, it was sold.

The growing demand from the home market was met by expansion of the Cork and Dublin factories in 1893, and a controlling interest in three fertiliser companies was acquired between 1897 and 1899. By 1902,



General view of the sulphuric acid/fertiliser plant at East Wall, Dublin.

sales had reached approximately 120,000 tons, and following additions to plant and acquisition of an interest in two more companies, sales increased to 150,000 tons in 1928 and 178,000 tons in 1939. Although seriously affected by the war, a large modernisation programme was carried out in the post-war years, and in 1951 the first granular fertiliser plant in Ireland was opened in Dublin.

The Goulding group of companies to-day operate seven factories producing sulphuric acid and superphosphate, in addition to powder and granular compounds. These factories are situated at Belfast and Londonderry in Northern Ireland, and at Drogheda, Dublin (two works), Wicklow and Cork in the Republic of Ireland. In the production of sulphuric acid, the raw materials used per annum are approximately 12,500 tons sulphur, 4,700 tons spent oxide and 45,000 tons pyrites, making in excess of 100,000 tons 100% H_2SO_4.

In addition to the seven acid-producing factories, there is another works at Waterford where powder and granular compounds are manufactured. Two new factories have also recently gone into production, one at Marina, Cork, where powder and granular compounds are being made, and the second at Sligo, which is making powder compounds. A second stage of development in these new factories envisages the introduction of acid and superphosphate plants, with the possibility of doubling the capacity later as a third stage of development.

The Company's policy is designed to meet the growing demand for fertilisers in the home market. In this respect the impressive modernisation and expansion programme should ensure ample supplies of top quality fertilisers specially formulated to meet the requirements of Irish soils.

The Marina fertilizer plant at Cork.

SULPHUR IN ITALY

THE reduction in September 1957 of the world price of sulphur, which was the direct result of the rapid expansion of sales by Mexican producers at the expense of the United States Frasch sulphur industry, has seriously impaired the prospects of the Italian sulphur industry. The ability of the Ente Zolfi Italiani to sell competitively in selected markets was, moreover, weakened by the continued slump in sea freights which narrowed the freight advantage of Italian sulphur. The limited resources which the Italian Government has placed at the Ente Zolfi Italiani's disposal for the purpose of subsidising the export price of Italian sulphur are thus being severely strained. To alleviate the position the Sicilian Parliament approved a draft law authorising the Ente Zolfi Italiani to guarantee the sulphur producers a minimum profit. This has been submitted to the National Parliament and it may be expected to come up shortly for discussion in the appropriate Committee. The Sicilian mining industry itself went through another crisis in mid-December when, because of extensive wage arrears, miners threatened an industry-wide stay-down strike. The timely passage of the draft law enabled the mine owners to secure financial relief which averted the strike.

Production of fused sulphur during the period January to the end of November 1957 totalled 162,030 metric tons, fractionally ($\frac{3}{4}$%) more than in the preceding year. The present rate of production of fused sulphur is about 15,000 tons monthly, a level substantially below the corresponding output of crude sulphur ore ;

the balance is used in the natural state for the manufacture of sulphuric acid. In addition to developments in Sicily, the use of sulphur ore and sulphur concentrates containing up to 50% sulphur is growing in importance on the Italian mainland. Sulphur ore mining in Central Italy is expanding and the discovery of the 10 million ton high grade sulphur ore deposit near Pomezia—about 25 miles north of Rome—may be expected to enhance its status. A consortium of three companies—Olin Mathieson Chemical Corporation, Società Italiani Squibb and Società Rumianca—has been formed and this group plans to exploit the deposit with a view to producing sulphuric acid. It has been mooted that a fluid bed roasting plant will be installed and that the output will be used in the manufacture of polyphosphates and other detergent intermediates.

Domestic sales of fused sulphur up to the end of November 1957 totalled 108,885 metric tons, approximately the same rate as in the preceding year. The Italian rayon industry, thanks to its competitive state, has been relatively little affected by the world-wide recession in this industrial sector and it is now reported that SNIA Viscosa plans to instal two viscose rayon plants—one for filament and one for staple fibre—at Trieste, where special advantages on account of taxes and duties may favour economics of production.

Exports

Despite the exceptionally high level of exports early in 1957—the highest rate since the war—the year's total to the end of November only amounted to 130,517 metric tons. The principal outlet has, as in the past, been France which accounted for 24% of total export deliveries; Israel (17½%), Tunisia (15%), and the U.S.S.R. (15%) were other major users of Italian brimstone. In all some 17 countries were supplied*.

General view of the Gibellini Mines; the site of Professor Roma's sulphur refining pilot plant.

Sulphur Stocks

On November 30th 1957 stocks of Italian sulphur held in Ente Zolfi Italiani's warehouses totalled 203,336 tons, which was represented by the following grades :—

	metric tons
Gialla Superiore	21,277
Gialla Inferiore	81,054
Buona	68,903
Corrente	32,102
Total	203,336

In the twelve-month period up to the 30th November 1957 sulphur stocks declined about 82,400 metric tons. This reduction of 29% compares with a decline of 62,000 tons (18%) in the preceding year. It is significant, however, that since the end of September there has been very little change—less than 700 tons—in stocks which emphasises the difficulties which have beset Italian exports since the reduction of world prices.

* For further details see Statistical Appendix.

NORTHWEST NITRO CHEMICALS LIMITED

THE new $22 million fertiliser plant of Northwest Nitro Chemicals Limited at Medicine Hat, Alberta, has within a year of commencing operations established itself as a major supplier of high grade nitrogenous and compound fertilisers to local and U.S. markets. The company owes its origin to the foresight of New British Dominion Oil Company Limited, who following the discovery with Midcon Limited, of attractive gas reserves in 1954 at Etzikom, near Medicine Hat, were joined by the Commercial Solvents Corporation of New York to manufacture fertilisers which would primarily be based on natural gas.

Financial backing was secured for the company, from both U.S. and Canadian sources, and of the first $1 million subscribed in cash for preferred stock, Commercial Solvents purchased 66% and New British the remainder. Through Eastman Dillon, Union Securities & Co., $9 million was raised by public issue of debentures and common stock, and $12 million was obtained from the sale of first mortgage bonds. Once the site of the new plant at Medicine Hat had been established, construction work—under the control of Ford, Bacon & Davis Inc., as main contractors for the whole project—went ahead rapidly and the plant was completed towards the end of 1956.

One of the principal features of the plant is the application of the St. Gobain process for the production of mixed fertilisers—notably diammonium phosphate — incorporating the phosphoric acid process developed by St. Gobain in conjuction with Union Chimique Belge. This is the first time that this process has been installed in North America.

Phosphoric Acid

The process in view of its simplicity shows favourable operating costs. Previously acidulation of the phosphate rock has required a number of reactors, which in the St. Gobain/Union Chimique Belge process have been reduced to a single stage. Phosphate rock requires a longer period of acidulation in this process but this is more than compensated by the reduced handling and the elimination of the need to grind the rock as finely as in conventional plants. The process also enables the uncalcined western phosphate rock, with its high organic content to be used as, although foaming is but slightly reduced, its nuisance is effectively checked.

The single digesters are capable of producing 62½ tons per day H_3PO_4 so that, with two units installed the company can take the best advantage of changes in demand for fertilisers. The individual units can be closed down for as long as three days without any alteration of the characteristics of the gypsum crystals appearing.

General view of the sulphuric acid/fertilizer installations at Medicine Hat, Alberta.

Ammonia and Sulphuric Acid

The granulation unit, which requires sulphuric acid for blending and ammonia for neutralizing, obtains its supplies from integrated plants built by the Kellogg Company of Canada, Ltd. The sulphuric acid plant of the Leonard Construction Co.'s design, has an annual capacity of 128,700 tons (100% H_2SO_4) contact acid and uses local recovered sulphur. The acid plant was the first to be completed, it was operating on a limited scale in October, 1956, and was closely followed by production from the nitric acid, ammonium nitrate, anhydrous ammonia, and ammonium phospate units.

The ammonia plant is based on the synthesis of natural gas and nitric acid requirements are met from a unit which employs the process developed by the Chemical and Industrial Corporation.

Raw Materials Requirements

The company's policy has been to enter into long term agreements for the supply of raw materials in order to minimise any adverse effects caused by fluctuations of the market and weather. Natural gas supplies are being obtained under contract from the South Alberta Pipelines, Ltd.—a joint company formed by New British Dominion and Midcon Companies —who operate the 10", 45 mile pipeline from the Etzikom gas field. Seven gas wells have already been established and total sales of natural gas contracted for the first fifteen years already amount to 92 thousand million cubic feet. Supplies surplus to Northwest Nitro's requirements are made available to the town of Medicine Hat.

The establishment of the natural gas cleaning plant by the British American Oil Company at Pincher Creek, provided an economic and favourably situated source of low cost sulphur. A five year contract gives Northwest Nitro Chemicals security of supplies at an annual rate of 44,000 tons high purity sulphur. Phosphate rock supplies originate from the J. R. Simplot Company in Idaho. The mine's inaccessibility in winter has made it necessary to build sufficient storage capacity to provide one year's supply. This has also involved the installation of additional rock handling facilities as it entails the build-up of the stockpile during the three summer months.

When in full operation it is estimated that about 410 tons per day of phosphate rock, and 140 tons per day of sulphur are required. Phosphoric acid production absorbs 3/5th of the daily sulphuric acid output of about 530 tons while the remainder is used in the production of ammonium sulphate phosphate fertilisers.

Fertilisers

The plant has a capacity to produce annually 140,000 tons of fertilisers and the flexibility of plant design enables varying grades of the different fertilisers to be produced. Annual design capacity for ammonium phosphate (11-48-0) is 65,000 tons, ammonium phosphate sulphate (16-20-9) 42,000 tons and ammonium nitrate 35,000 tons (35½% N). To this range of products was added early this year ammonium nitrate phospate (27-14-0). Output is valued at about $11 million annually and the company has a large distributive organisation in the Prairie Provinces, with Harrisons and Crossfield (Canada) Limited as exclusive sales agents.

Summary

The fertiliser plant of Northwest Nitro Chemicals represents an initial and significant stage in the utilisation of the vast Canadian resources of natural gas and the rapidly mounting supplies of by-product sulphur. Producing a range of nitrogenous fertilisers, principally for the western Canadian market, the company has also had considerable success in establishing a market in the North-west United States. In view of the present relatively low utilisation of ammonia capacity in the Western States this penetration reflects the high degree of competitiveness of the company's product which is partly based on favourable location to raw materials and markets, while the plant design ensures favourable economics and a rapid change in types of fertiliser production to meet the varying needs of the market.

Current Events

UNITED KINGDOM

Simon Carves Ltd. — Plant Construction Progress

The *Simon Group's Autumn Review* gives an account of recent completions of sulphuric acid plants and installations, principally in the United Kingdom and the Commonwealth. Two new sulphuric acid plants, each with a daily capacity of 50 tons (100% H_2SO_4), have been commissioned; one for Messrs. R. & J. Garroway, at Netherfield, Glasgow, and the other for the Indian Iron and Steel Company at Hirapur. Good progress is reported on work in hand in the United Kingdom; the 140 tons per day plant for Fisons Ltd., at Immingham, the 175 tons per day of Laporte Titanium Ltd., at Stallingborough, and the sulphur recovery plant based on acid gas for British Petroleum Refinery at the Isle of Grain, Kent, are all expected to be in service in the spring of 1958.

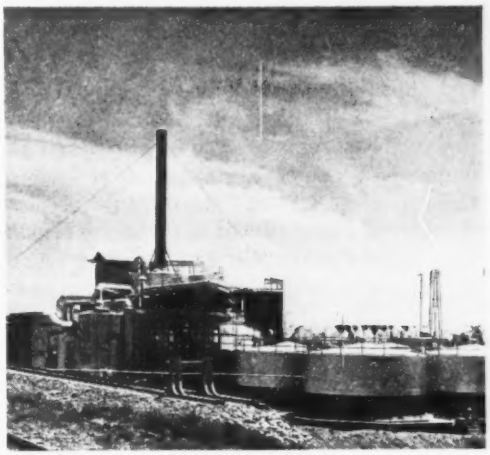
In the Southern hemisphere erection of the following plants is proceeding satisfactorily:—

		100% H_2SO_4
Mary Kathleen Uranium Ltd.	Australia	110 tons
Kiwi Fertiliser Company	New Zealand	110 tons
Bay of Plenty Fertiliser Works ...	New Zealand	110 tons
Southland Co-operative Phosphate Company ...	New Zealand	110 tons
African Explosives & Chemicals Industries (Rhodesia) Ltd.	Rhodesia	182 tons

Widespread interest has been aroused by the 100 tons per day wet-contact plant for the Shell Company of Australia Ltd., at Geelong. This is the first unit in Australia to make use of sulphur in oil refinery gases. The resultant acid is to be supplied to the adjacent works of Cresco Fertilisers Ltd. In the Union of South Africa, Fisons (Pty.) Ltd. is building, as part of a major fertiliser project, a 230 tons per day sulphuric acid contact plant at Sasolburg.

Work has started on the installation of tail gas mist precipitators at the anhydrite acid plant of United Sulphuric Acid Corporation at Widnes, and on three mist precipitators for British Titan Products Company Ltd., at Grimsby.

Messrs. Huntington, Heberlein & Co. Ltd.* report that the wet contact acid plant for the North Thames Gas Board at Beckton is virtually completed and that all the materials for the six Herreschoff furnaces, for the Rhodia sulphuric acid plant in South Africa, have now



Blyvooruitzicht Gold Mines Ltd. (Union of South Africa). The 150 tons per day sulphuric acid plant recently completed by Simon-Carves Ltd.

been shipped. Other work by this company includes the erection of an acid concentration plant for the Government of Pakistan, and the installation of a ceramic filter on the South-Eastern Gas Board's Simon-Carves Kachkaroff acid plant at Phœnix Wharf.

British Chrome and Chemicals Ltd.

Following the extensive re-organisation and expansion in 1956 of the production facilities for sodium bichromate and chrome chemicals, *British Chrome & Chemicals Ltd.*, have now decided to expand the sulphuric acid capacity of the Eaglescliffe Chemical Works. An order has been placed with the Power Gas Corporation Ltd. for the erection of a 100 tons per day (100% H_2SO_4) sulphuric acid plant based on the design of Chemiebau Dr. Zieren and the use of brimstone. Output will be in the form of 95% and 70% acid.

Formerly the Eaglescliffe Chemical Co. the company's present name was registered after the purchase of John & James White Ltd.

* See also page 34, Norway

in 1953. The parent company, British Chrome & Chemicals (Holdings), has now merged with Brotherton & Company of Leeds. The latter is a leading producer of sulphur dioxide and sulphites and the group now becomes one of the major sulphur users in the United Kingdom.

CANADA

New Sulphur Recovery Project

The *Texas Gulf Sulphur Company* of New York, in co-operation with Devon-Palmer Oils Ltd., Canadian Shell Explorations Ltd., and Shell Oil Company, all of Calgary, Alberta, is planning large-scale elemental sulphur production based on sour natural gas discovered by the Shell Oil Company of Canada in the Okotoks gas fields district.

Devon Palmer Oils Ltd., who are responsible for the production and gathering system of the gas, report the following analysis :—

	%
Carbon Dioxide	10.72
Nitrogen	1.32
Hydrogen Sulphide	34.39
Methane	52.44
Ethane	0.53
Propane	0.14
Others	0.46

It is estimated that from the processing of each one million cubic feet of gas there will be 480,000 cu. ft. of residue gas for sale, 11.6 long tons of sulphur and 2.6 barrels stock tank condensate 35° API. The gas will be supplied at a daily rate of 31 million cu. ft. from ten wells, seven of which are owned by The Devon-Palmer Group.

Texas Gulf Sulphur will operate the gas cleaning and sulphur recovery plant, and will utilize a process similar to that employed at the Worland, Wyoming, plant. Monoethanolamine is to be used as the absorbent and is normally diluted to 15%-25% of amine by weight. The solution is passed countercurrently to the sour gas in a tray type absorber, and one pass is generally sufficient to produce a gas with an H_2S content of less than one grain per 100 cu. ft. which makes it acceptable for piping. Stripping the amine solution releases the concentrated H_2S and partial combustion and catalysis in a Claus type kiln produces elemental sulphur and permits heat recovery in a waste heat boiler. Capacity will be 360 tons per day of high purity brimstone, and the gas cleaning plant will produce 14.1 million cu. ft. of sweet gas and 78 barrels of liquid petroleum condensate. The dry gas is to be sold either to a public utility Company or to an exporter of natural gas to the United States, possibly the Southern Alberta Gas and Pipeline Company, which plans to run a 1,300-mile line direct to San Francisco.

Brown & Root Inc., of Houston, Texas, are in charge of construction and site preparation work has been commenced. The plant was originally designed to enter production towards the end of 1958, but it is understood that the recent reduction of U.S. price of Frasch sulphur may modify this date.

INDIA

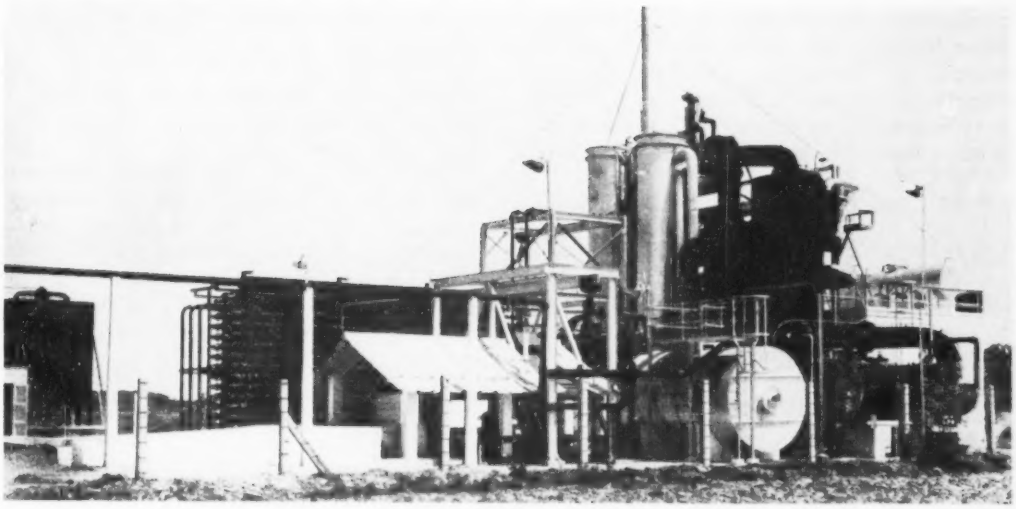
Sulphur State Trading

Since 1st January 1958, State-controlled purchasing of sulphur is believed to have taken the place of licencing and quantitative import control. The new State Trading Department at New Delhi, which already deals in soda ash and caustic soda, is expected to conclude contracts with individual sulphur suppliers on the basis of requisitions by the industry. Distribution and sales at controlled prices to individual users is expected to be effected through the intermediary of the former major importers and distributors, most of whom are the agents of the principal sulphur producers.

Acid/Fertiliser Plant in Operation

Shaw Wallace & Company Ltd. have recently commissioned a sulphuric acid/fertiliser plant in Madras. Built by Simon-Carves Ltd., of Cheadle Heath, Cheshire, the brimstone burning acid plant has a capacity of 20 tons per day, and the superphosphate plant is capable of producing 100 tons per day. Since the war the rapidly growing needs of the Indian fertiliser and heavy chemical industries has prompted intensive competition between plant makers. Simon-Carves Ltd., who have built, on average, one plant a year in India during this period, have now installed there capacity of over 100,000 (100% H_2SO_4) tons per annum.

At present there are 38 sulphuric acid plants in operation in India with an annual capacity of one-quarter million tons (100% H_2SO_4).



General view of Shaw Wallace and Co. Ltd's. sulphuric acid plant in Madras.

U.S.A.

New Sulphuric Acid Project

The General Chemical Division of the *Allied Chemical and Dye Corporation* has recently acquired a 16-acre site adjacent to the Shell Oil and Texas Companies' refineries at Anacortes, Washington. Site preparation work has commenced, and final designs are now being completed for the erection of a sulphuric acid plant which will have facilities for the burning of refinery sludge acid and hydrogen sulphide gas.

The Texas Company's refinery, known as the Puget Sound Works will, when completed, produce a wide range of petroleum products for the growing North-West and West coast markets. It will include a fluid catalytic cracker with a daily capacity of 18,000 barrels, as well as catalytic reformers and polymerisers and alkylation units. Shell Oil Company's refinery has a crude oil throughput of 50,000 barrels a day, and is based predominantly on Western Canadian and some Californian crudes. At present an alkylation unit is being installed at the cost of several million dollars. Built by Fluor Corporation, and due on stream mid-1958, the unit will have a daily capacity of 24,000 barrels crude alkylate, sufficient for blending into 800,000 barrels 115/145 aviation fuel. The process involves the mixing under refrigerated conditions olefins and isobutane in the presence of a sulphuric acid catalyst. This project is another example of co-ordinating the useful disposal of sludge acids and sulphur values in refinery gases with the assurance of adequate low cost sulphuric acid supplies, a practice which is becoming increasingly popular in the oil industry.

Biological Application of Sulphur

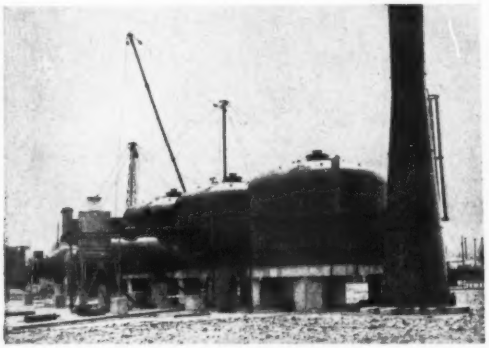
Following two years research by scientists at the University of Illinois, results have recently been published on the effect of the addition of sulphur to sheep's feed. It has been found that the addition of raw sulphur to the animals diet prevents shrinkage of wool; sulphur appears to find its way into wool fibres to promote the formation of a molecular bridge. The addition of about one part of sulphur in a hundred of the sheep's total feed intake is reported to have given the best results.

FRANCE

Sulphur Recovery Plant

Commissioned by Bozel Maletra, a leading sulphuric acid and chemicals producer, a sulphur recovery plant is currently being built by Power-Gas France at Port Jérôme, near Le Havre, adjacent to the oil refineries of Esso Standard and Socony Vacuum. Designed in

co-operation with Comprimo N.V., of Holland, under licence to Bataafsche Petroleum Maatschappij, of The Hague, the plant is being completely fabricated in France, and will have a daily capacity of 60 tons high purity brimstone. Bozel Maletra are understood to have



Bozel-Maletra. Sulphur recovery plant under construction.

earmarked the new sulphur output for use in their Petit Quevilly works, where recently acid plant modernisation included the installation of additional brimstone burners.

GERMANY

Native Sulphur Deposits

In the course of oil exploration in the Emsland, North-West Germany, sulphur deposits have been encountered at a depth of 2,000-2,500 feet. Their extent has not so far been determined and the Gewerkschaft Barbara in Salzgitter is making further investigations.

ITALY

Expansion of By-Product Acid Capacity

At the *Condor Oil Refinery* located on the outskirts of Milan, the completion of a new sulphuric acid plant raises the installed daily capacity to 90 metric tons (100% H_2SO_4). The oil refinery uses Middle East crudes containing 1½%-2½% sulphur, and to obviate the hydrogen sulphide in the gas streams causing air pollution, it is extracted and converted by the wet-contact process into sulphuric acid.

The Company's first acid plant was commissioned in August 1955; it has a daily capacity of 30 metric tons, and was built by Oronzio de Nora of Milan on the Monsanto-de Nora patent.

The expansion of the refinery, which included raising the throughput of the catalytic cracker to 15,000 barrels per day, entailed extra desulphurisation facilities to recover the additional sulphur values. The new acid plant which was completed in October 1957, has a rated capacity of 60 tons per day (100% H_2SO_4), but is otherwise identical in design and layout. The resultant sulphuric acid is very pure—practically water white—and its manufacture represents a conversion efficiency of about 95% of the sulphur content of the gas, which assays as follows:—

	Weight %
H_2S	94-92
H_2O	1-2
Hydrocarbons ...	4-6
NH_4	0.8-0.9

It is believed that this installation is the only one of its kind in Italy to-day, although two other oil refineries are reported to be planning H_2S extraction from gases, but recovery is to be in the form of brimstone.

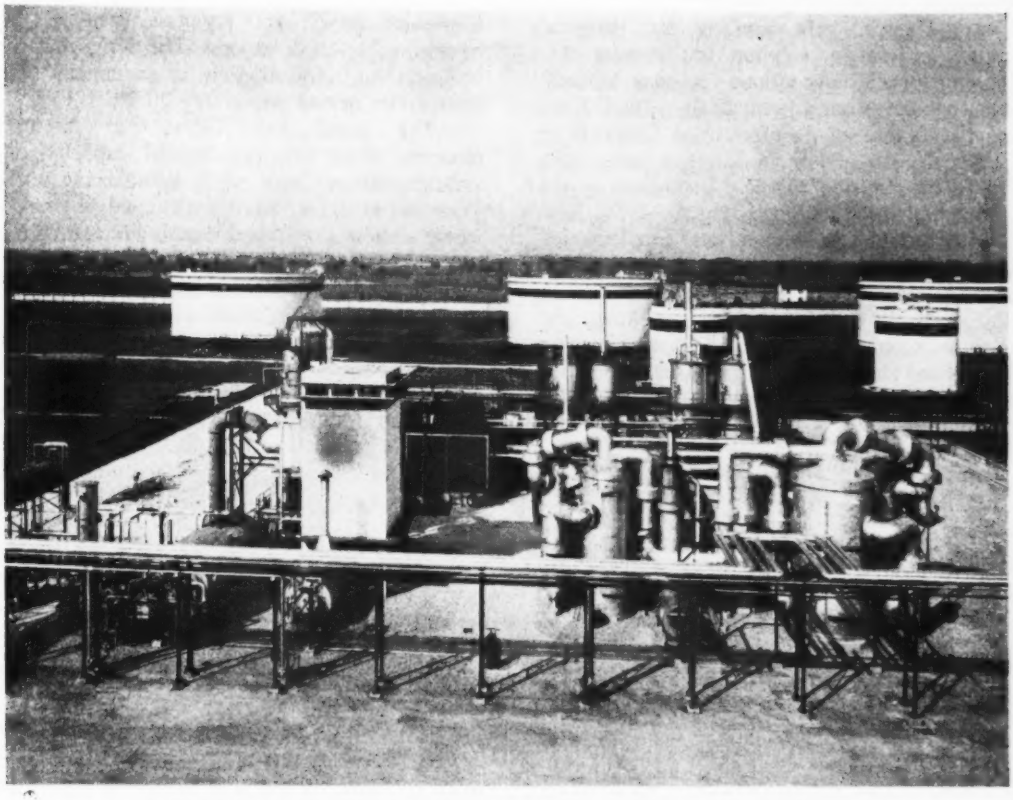
NORWAY

Greater Flexibility at Acid Plant

Messrs. Norsk Spraensgstofindustri A/S have commissioned Messrs. Huntington, Heberlein & Co. Ltd., London, to instal a Glens Falls brimstone burner at their Oslo sulphuric acid plant. This will duplicate the existing pyrites roaster and allow the company to take advantage of the cheaper source of sulphur supply. Norsk Spraensgstofindustri A/S, who are explosives and chemical manufacturers, produce annually about 15,000 tons (100% H_2SO_4), although converter capacity is somewhat greater.

New Pyrites Production

A project to reactivate the Kongsberg Silver Mines, situated in the Saggrenda district of East Norway, is under consideration by *Lysaker Kemiske Fabrik A/S*. The Company is reported to be arranging to lease the mines from the Government for a period of 50 years. The mines have been pumped dry and development work is in hand and if the survey results are satisfactory regular mining should start in the spring of 1958. Messrs. Lysaker Kemiske Fabrik are sulphuric acid makers whose installations comprise a contact unit and a Petersen tower with combined capacity of 45,000 tons



Condor Oil Refinery. Wet contact acid installations built by Oronzio de Nora of Milan.

(100% H_2SO_4) of which about 55-60% is utilised. At present the Company manufactures sulphuric acid based predominantly on bought-in Norwegian pyrites, supplemented by a small quantity of brimstone.

The favourable location of the Kongsberg mine in relation to the acid plants at Lysaker near Oslo, offers a relatively cheap source of sulphur supply. In the assessment of the project the economics of acid manufacture are an important factor and, if the Company's Board favour the project, the planned annual output of 7,000 to 10,000 tons pyrites in the form of flotation concentrate, assaying about 45% S, 40% Fe and free from Arsenic, would meet about one-half of the Company's sulphur needs. Although relatively small, the development underlines the trend everywhere of keener costing of raw materials supply.

SPAIN

Sulphur Production — 1956

The first full year of operation of the new sulphur recovery plant of Real Cia Asturiana de Minas at Hinojedo was primarily responsible for the 29% rise in domestic Spanish sulphur production to 52,699 metric tons. Individual productions in 1955 and 1956 are as follows:—

	1955	*1956*
Coto Minero de Hellin S.A. ...	2,944	2,555
Cia Espanola Minas Rio Tinto S.A.	34,102	36,600
Azufres de Lorca S.A.	2,020	2,834
Real Cia Asturiana de Minas ...	1,018	10,299
La Industrial Quimica de Zaragoza	816	392
	40,900	52,669

Production of brimstone by the Cia Espanola Minas Rio Tinto S.A., Spain's most important producer, has shown little change in recent years and, output arises from the smelting of pyrites at its Orkla plant at Rio

Tinto, Huelva. In view of the marginal nature of native sulphur ore mining and refining, producers continue receiving Government subsidy from an equalisation fund. Even so, the failure of La Industrial Quimica de Zaragoza to treat low grade ores economically led to the decision to cease operations in the summer of 1956. Since January 1956, Real Cia Asturiana de Minas has supplemented indigenous brimstone supplies from their new plant at Hinojedo. This is based on a process developed by the American Smelting and Refining Company, whereby SO_2 contained in pyrites and zinc blende roaster gases is absorbed in cold dimethylaniline solution from which concentrated SO_2 is released. The plant is designed to yield 260 tons per day SO_2, of which 90 tons will be produced in liquid form and the balance, after reduction with producer gas, as elemental sulphur at a daily rate of 70 tons. The entire output is consumed by domestic users.

Empresa Nacional Calvo Sotelo. General view of nitrogenous fertilizer plant. Sulphur extraction and recovery unit, centre.

Sulphur Recovery from Synthesis Gas

Construction work at the nitrogenous fertiliser plant of *Empresa Nacional Calvo Sotelo de Combustibles Liquidos y Lubricantes S.A.* at Puentes de Garcia Rodriguez, La Coruña, is nearing completion. Synthesis gas containing 83% $CO+H_2$ is obtained by the combustion of lignite in the presence of oxygen by the Koppers-Totzek process; nitrogen is generated in a Linde installation and ammonia synthesis is based on the Casale process. The designed rate of operations is 100,000 tons per annum calcium ammonium nitrate containing 20.5% N_2.

The installations include a sulphur recovery plant with an annual capacity of about 1,500 tons high purity liquid brimstone. This is a Claus Kiln with catalytic converter section of standard design which obtains its H_2S feed from a hot potassium carbonate desulphurisation unit installed to purify the synthesis gas stream containing 1.6% H_2S or about 1,000 grains/100 cu. ft. This plant, built by Messrs. Koppers of Essen in 1954 has a daily (24hrs.) treatment capacity of 10.3 million cu. ft. gas. To desulphurise the gas which is under pressure and from which benzol has been removed, it is washed counter-current in a bubble cap scrubbing column with a potassium carbonate-bicarbonate solution which absorbs H_2S and some CO_2. Foul potassium carbonate solution is regenerated in a distillation column under vacuum and at a temperature of 70°C, but owing to side reaction of hydrogen cyanide which is also extracted, about 15% of the scrubbing solution is lost and has to be replaced.

The extracted acid gas containing 75-80% H_2S and some CO_2 provides, after cooling and condensation, the feed for the Claus kiln. Heat recovery provides most of the steam require-

ments and it is believed that under prevailing conditions the operations of the sulphur extraction and recovery process will be profitable.

By-Product Acid Project

At the oil refinery of the *Compania Espanola de Petroleos S.A.* in the Canary Islands by-products are to be used for fertilizer manufacture. The refinery located at Santa Cruz de Tenerife has a daily crude oil throughput of 35,000 barrels and a catalytic cracker is capable of treating one-tenth of this total. Hydrogen sulphide extracted from the refinery gases will be fed to a 250 tons per day contact sulphuric acid plant. Synthesis gas, produced by the Texaco process, will be used for ammonia manufacture.

The plant which will cost about $16 million is to have an annual capacity of 110,000 tons ammonium sulphate; the project has been approved by the Instituto Nacional Industria.

LEBANON

Fertiliser Manufacture

The new superphosphate plant of the *Lebanon Chemicals Company* is due to start operation in February. It will have an annual capacity of 48,000 tons and will meet Lebanon's growing phosphatic fertiliser requirements; these are met wholly by imports. Located at Batroum, North Lebanon, the plant will include a 50 tons per day contact sulphuric acid unit, based on the use of brimstone; this is to be purchased from both U.S. and Italian sources, while phosphate rock is to be bought from Jordan. The superphosphate plant provides for the by-product manufacture of sodium silico fluoride.

JAPAN

New Pyrites Ore Body Discovered

Already one of the two largest pyrites producing deposits in Japan, the Yanahara mine of the *Dowa Mining Company*, is to have its output further increased by the addition of a newly-discovered and contiguous deposit. Although Yanahara was discovered in 1882 it does not appear to have been exploited until 1915 when a small output of 21,400 tons was recorded. This has since grown to 482,000 tons in 1956.

The new body, discovered in 1955, is on the lower side of a major fault and believed to be the continuation of the existing deposit. Plans have been laid for the exploitation of this massive deposit at a monthly rate of 60,000 tons and development work, which includes the sinking of a 600 metre shaft, has already commenced. To be completed by 1960, it is reported that the output will be as follows:—

	% *S*	*tons*
Pyrite	48	660,000
Pyrrhotite	35	36,000
Cu-pyrite	42	24,000
		720,000

The production will be sold to sulphuric acid plants in the middle west of Japan and in Kyushu; these account for about 25% of the total sulphur demand in Japan.

CHILE

By-Product Acid Production Plans

In conjunction with the development work at their new copper smelter at Paipote, Empresa Nacional de Fundiciones are planning major by-product acid installations. The present smelter, which started operations in 1952, consumes sulphide copper ore and concentrates at a daily rate of 400 tons. The matte obtained from the reverberatory furnace is converted into blister copper at a rate of 15,500 metric tons per annum following a capacity expansion last year. Plans for a contact sulphuric acid plant were nearing completion last May when the only detail unresolved was whether it is to be based on the use of the roaster gases from the copper concentrates which have a high SO_2 content or on those of the convertor which have a low content. The designed capacity is 45 tons of 98% acid and plant operation is due to start in 1958.

The company's plans also foresee the erection of a second copper smelter at Papudo — 180 kilometres north-east of Santiago in central Chile. This is to have a smelting capacity of 150,000 tons of copper concentrates, resulting in the manufacture of 27,000 tons blister copper per annum. The joint electrolytic refinery is to treat this as well as the blister copper output from Paipote. Plans provide for the construction of a contact sulphuric acid plant with a daily capacity of 20 tons of 98% acid, with provision to double this capacity. The intention to roast the copper concentrates

in Herreshoff furnaces will facilitate the supply of SO_2 rich gas for conversion into sulphuric acid.

The output of sulphuric acid at Paipote is to be used exclusively for leaching copper oxide ores from which cement copper is to be derived. The Papudo output, on the other hand, is to be used in addition to copper leaching for supply to the electrolytic refinery and also to the petroleum, rayon, metallurgical and various other industries in central Chile. The decline in copper prices since these plans were formulated may be expected to delay the construction of the second smelter but the Paipote scheme is understood to be unaffected.

BULGARIA

Acid/Fertiliser Plant

Built with Russian financial and technical assistance, the new chemicals and fertiliser installation at Dimitrovgrad is Bulgaria's first major chemicals plant. The integrated sulphuric acid unit which started operations in June 1957, is based on pyrites of domestic origin. In 1956 Bulgarian sulphuric acid output rose 48% to 28,600 metric tons (100% H_2SO_4) as compared with 19,400 tons in 1955. The new plant increases the country's production capacity by about 50% to 40,000 tons per annum. The bulk of this acid output is now used in a 100,000 tons per year superphosphate plant which started production in the autumn. Expansion of fertiliser manufacture is under way, and it is reported that in addition to doubling the nitrogenous fertiliser plant to produce 200,000 tons annually, the superphosphate plant will be expanded over the next four years to 400,000 tons capacity. These plant facilities make Bulgaria self-sufficient in nitrogenous and phosphatic fertilisers, and there is the possibility that Bulgaria may become an exporter to neighbouring countries.

POLAND

Sulphur Mine in operation

On 4th December, Miners' Day, the first sulphur mine in the history of Poland was commissioned in Piaseczno, near Tarnobrzeg, Rzeszow district. The opening ceremony was performed by the Minister of the Chemical Industry, Mr. A. Radlinski, accompanied by the Czechoslovak Ambassador, Mr. K. Vojacek, and the discoverer of the Tarnobrzeg sulphur deposits, Professor S. Pawlowski.

In order to reach the deposits, which lie beneath an overburden of 200-250 feet, the Piaseczno miners had to remove over 400 million cubic yards of earth and drain away over 1,430 million gallons of water from the mine. In 1958 the Piaseczno mine* is to yield 50,000 tons of ore, and in 1960 about one-half a million tons.

U.S.S.R.

Transportation of Sour Natural Gas

According to United States sources Russian engineers have successfully overcome the problem of transporting heavily-contaminated sour natural gas by carrying it in an asbestos-cement pipeline from the Bashkiria Ishimbai field—Russia's "Second Baku." Experience of this type of piping has been obtained in its previous use for gas and water. The first use is reported in 1943 on a 12-mile section of the Burguruslan-Kuibyshev pipe where gas is piped at a pressure of 88 psi and on a five-mile section of the pipeline from Andizhan fields which operates at 45 psi. The new pipe which is to extend for 19 miles is reported to operate at pressures up to 150 psi while the thickness and volumetric weight of the walls are about 20% greater than for the asbestos-cement waterpipe. It is not subject to soil corrosion and the low co-efficient of expansion obviates temperature deformation and stress while the cost of manufacture is about one-third to one-half that of iron or steel pipe.

* For full details of project see Issue No. 17, Page 26.

Statistical Appendix

Mexico

SULPHUR EXPORTS — July-September 1957 — metric tons

	Pan American Sulphur Co.	Gulf Sulphur Corp.	Total 3rd Qtr.	Total Jan.-Sept. 1957
U.S.A.	83,652	27,650	111,302	337,232
United Kingdom	29,344	3,052	32,396	69,177
France	17,403	—	17,403	54,529
Australia	7,723	—	7,723	49,577
Germany	3,687	—	3,687	5,878
Holland	10,559	—	10,559	30,246
New Zealand	6,097	—	6,097	25,662
Belgium	5,589	—	5,589	17,932
Tunisia	4,890	—	4,980	8,237
South Africa	23,074	—	23,074	23,074
Israel	—	10,163	10,163	10,163
Total	192,109	40,865	232,974	631,707

United States

SULPHUR EXPORTS

Long Tons

	3rd Qtr. Total	Jan.-Sept. Total		3rd Qtr. Total	Jan.-Sept. Total
Canada	121,973	250,981	Switzerland	7,859	29,675
Cuba	3,800	12,443	Yugoslavia	—	2,000
Argentina	6,922	29,331	Iran	4,500	4,500
Brazil	12,334	68,073	India	10,966	73,386
Uruguay	—	3,050	Taiwan	1,673	1,673
United Kingdom	59,002	188,981	Algeria	—	8,271
Austria	5,500	14,700	Morocco	3,000	7,000
Belgium	16,200	59,900	Tunisia	—	4,000
Canary Islands	1,500	5,489	Australia	27,385	51,510
France	25,500	91,600	New Zealand	5,900	37,555
Finland	9,725	18,545	Union of South Africa	13,000	64,350
Netherlands	4,500	7,000	Other Countries	2,229	17,424
Sweden	2,000	7,950			
West Germany	14,300	47,845	Total	359,768	1,107,232

SULPHUR PRODUCTION

	PRODUCTION		STOCKS		EXPORTS	IMPORTS
	Frasch	Recovered	Frasch	Recovered		
1953	5,155,342	337,099	3,022,486	107,344	1,241,536	
1954	5,515,543	357,400	3,228,020	109,066	1,647,011	1,104
1955	5,743,344	401,650	3,181,198	120,267	1,595,419	38,317
1956	6,427,229	480,980	3,936,450	119,446	1,651,597	203,300
July	621,130	43,400	3,493,942	115,246	136,048	15,036
August	597,699	42,500	3,636,730	115,047	188,240	15,347
September	524,370	42,000	3,680,784	117,759	138,784	28,440
October	529,056	40,750	3,705,626	118,937	192,332	23,814
November	514,772	39,580	3,852,028	119,157	95,003	19,254
December	534,088	43,750	3,936,450	119,446	148,367	20,644
1957 January	495,353	42,154	3,997,911	118,296	115,747	33,883
February	439,997	39,000	4,087,925	116,713	74,698	29,141
March	471,548	39,800	4,102,394	111,905	99,637	44,673
April	461,589	37,650	4,049,025	122,006	171,258	43,897
May	429,227	42,400	4,092,772	127,908	113,147	39,847
June	439,513	44,867	4,086,614	144,753	172,977	24,638
July	459,534	45,311	4,153,206	150,518	118,814	47,918
August	469,987	46,705	4,173,199	153,478	161,337	42,053
September	444,779	43,912	4,272,558	152,940	79,617	

Official Bureau of Mines Data (long tons of 2,240lbs.)

Italy

SULPHUR EXPORTS — January-November 1957 — metric tons

Algeria	1,410
Austria	3,675
Czechoslovakia	4,500
Egypt	8,432
France	35,150
Germany	1,000
Greece	1,200
Iran	990
Israel	22,245
Jugoslavia	7,024
Lebanon	660
Pakistan	7
Poland	2,500
Rumania	1,848
Sweden	1,000
Tunisia	19,720
Russia	19,156
Total	130,517

Cyprus

IRON PYRITES EXPORTS

Long Tons

	January-October 1957	1956
United Kingdom	151,105	178,960
Belgium	14,705	8,186
Czechoslovakia	14,179	—
France	115,409	163,997
Western Germany	201,546	202,534
Netherlands	88,395	189,519
Italy	53,102	44,591
Egypt	8,256	33,940
Other Countries	1	—
Total	647,738	821,727

CUPREOUS PYRITES EXPORTS

January-October

	1957	1956
Western Germany	75,022	67,329
Netherlands	66,145	41,603
Italy	71,869	62,100
Total	213,036	171,032

Norway

SULPHUR EXPORTS

Metric Tons

	January-September 1957	1956
Finland	12,298	25,013
Sweden	30,970	45,915
Denmark	947	564
Others	117	91
Total	44,332	71,583

PYRITES EXPORTS

January-September

	1957	1956
Poland	6,830	15,056
Czechoslovakia	18,529	22,029
East Germany	45,566	63,072
Sweden	82,075	110,235
France	2,058	14,756
West Germany	119,833	165,974
Netherlands	9,041	7,588
Denmark	48,805	60,645
Others	—	8,395
Total	332,737	467,750

Western Germany

CRUDE SULPHUR IMPORTS

Metric Tons

	January-September 1957
Italy	1,000
U.S.A.	37,530
Mexico	9,068
Chile	399
Total	47,997

PYRITES IMPORTS

January-September

	1957
Finland	37,929
Greece	2,755
Jugoslavia	23,618
Norway	139,047
Portugal	35,149
Sweden	5,276
Spain	496,708
Turkey	30,193
Canada	59,321
Cuba	62,235
Cyprus	322,188
Others	1,330
Total	1,215,759

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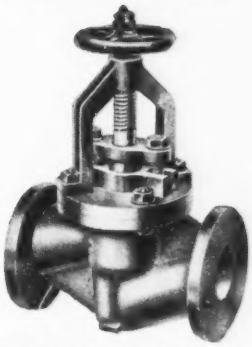

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SUP

SULPHUR

SPECIAL NUMBER 1957

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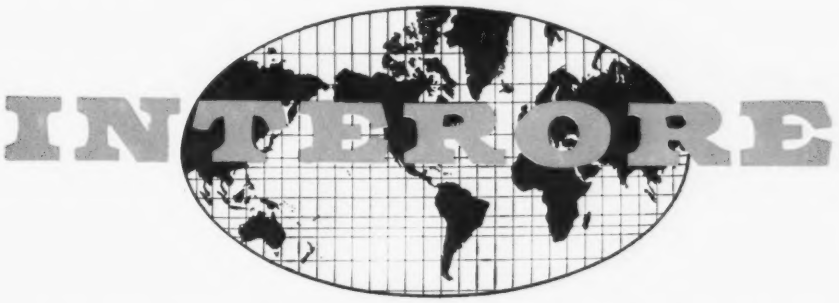

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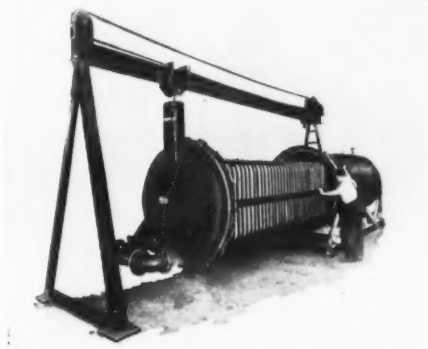

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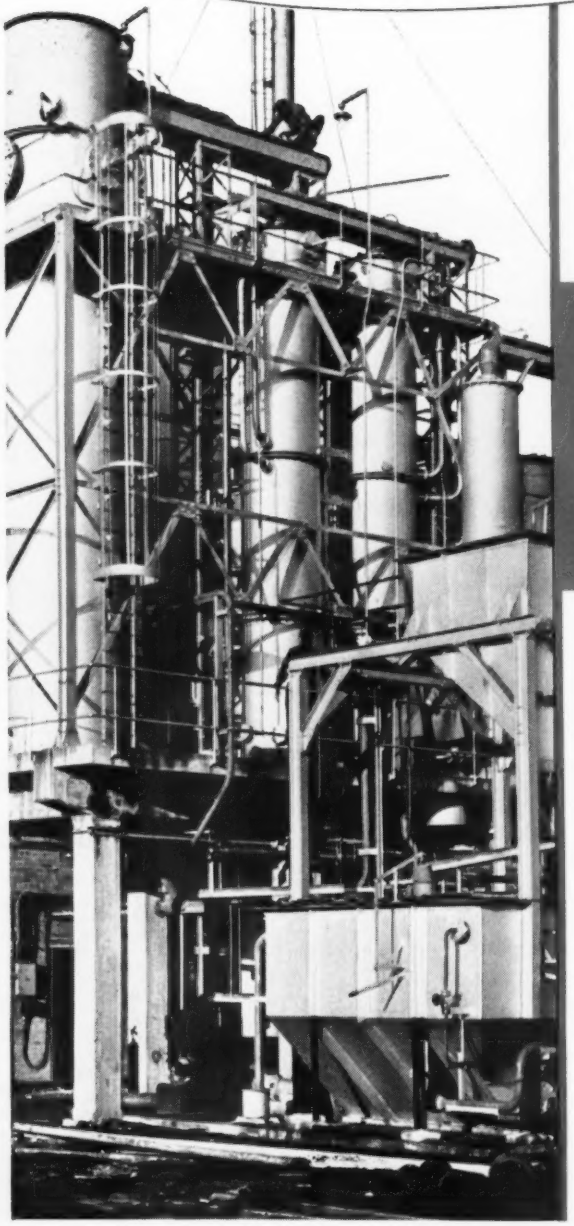

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TURBULENT LAYER ROASTING

THE BASF PROCESS AND PLANT

BASED on principles developed in the early 1920's by Dr. F. Winkler for the efficient and controlled gasification of coal, the industrial application of fluidised bed or turbulent layer roasting to metallic ores is essentially a post-war achievement. The technique was developed practically simultaneously in Germany by the Badische Anilin-& Soda-Fabrik of Ludwigshafen and in the United States by the Dorr Company of Stamford, Connecticut — the patentees of the Dorr Company FluoSolids System — and by the New Jersey Zinc Corporation. Both Dorr and New Jersey Zinc Companies were in the first place concerned with the metallurgical problems of efficiently roasting arsenical gold ores, copper ores, zinc blende, and other metallic ores although the Dorr FluoSolids process also rapidly made an impact on pyrites roasting notably in the U.S.A., Canada and South Africa. BASF—one of the largest sulphuric acid manufacturers in Europe and one of the pioneers in the development of the contact process—from the start focussed their attention on pyrites. Experiments, with pyrites fines and flotation concentrates which had been started in 1943, at the Leuna works, were brought to successful conclusion in 1950 with the operation at Ludwigshafen of a 36 tons per day (100% H_2SO_4) plant, which produced SO_2 gas from pyrites roasted in a turbulent layer. It ushered in a new era in pyrites roasting plant construction which in little over one-half century had advanced from lump burners to the multi-hearth furnaces, kilns and flash roasting installations each built to contend with the roasting problems of the great variety of size and quality of pyrites ores and concentrates.

The principle of fluidised bed process is to transmit a gas stream through a bed of fine solids at such a speed that this assumes the physical properties of a liquid. Intimate contact between solid particles and gas accelerates the chemical reaction. The gas stream may also contain several components one or more of which catalyse the reaction. Moreover, the fluid-like nature of the bed promotes temperature equalisation. In the BASF process the actual turbulent layer comprises almost completely roasted pyrites which is at roasting temperature, and maintained by a stream of cold air. It is replenished with pyrites by a controlled feed mechanism. Roasting is effected with slight excess of air, and may be represented by the following stochiometric formulae :—

$$FeS_2 + \tfrac{11}{4}\ O_2 \rightarrow \tfrac{1}{2}Fe_2O_3 + 2SO_2 + 205.5\ \text{Kcal.}$$
$$FeS_2 + \tfrac{8}{3}\ O_2 \rightarrow \tfrac{1}{3}Fe_3O_4 + 2SO_2 + 195.2\ \text{Kcal.}$$

In theory this yields a gas concentration of 16% SO_2, though in practice the gas stream contains 12-14% SO_2, which is of course quite adequate. The roasting process is highly exorthermic and temperature in the roasting zone could rise to over 1,600°C causing agglomeration of the pyrites charge and a breakdown of fluidisation. To prevent this, heat must be removed and in the BASF process this takes place indirectly through cooling elements in or above the turbulent layer or on the outer wall of the chamber. The heat removed in reducing operating temperatures to between 750° and 900°C—depending on the nature of pyrites—is recovered as steam. This method avoids the roaster gases being diluted by gaseous cooling media and ensures an effective exploitation of the heat of roasting.

Turbulent Layer Roaster

Figure 1 depicts schematically a BASF roaster. Air is propelled by a blower (1) through the chamber (6) and a close slotted grid (7) to the fluidised bed (8) which fills the lower part of the roaster and which receives its pyrites charge by the feed mechanism (2). Heat in excess of that required to maintain the turbulent layer at the correct temperature is removed by cooling devices, while the sensible heat of the roaster gas is recovered through a steam superheater (10) and a boiler (11). From the steam drum (13) hot water—under pressure from the generated steam—is pumped (14) in two parallel lines, on the one hand by way of the steam pipes in the turbulent layer and also through the boiler (11).

The resultant mixture of steam and water is fed back to the steam drum. After being separated from the circulating water, steam is heated to the requisite temperature in the superheater (10) whence it is distributed to the works. The residues are removed from

the roaster by way of a discharge pipe which is fitted with regulator flap. Residual

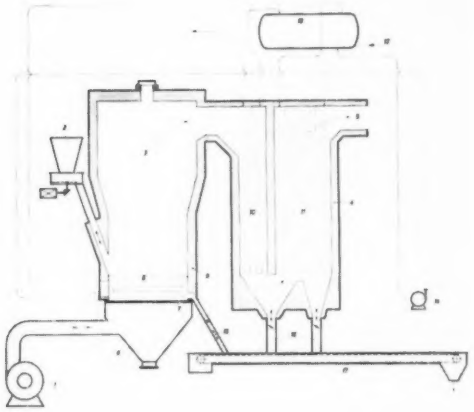


Fig. 1.

dust eliminated on the waste heat boiler (4) is discharged through twin tubes on to a travelling belt.

Furnaces with circular cross-section have proved more serviceable than square structures primarily on account of the better balance of thermal tensions in the ceramic linings and the avoidance of cracks in the brickwork.

Raw Materials

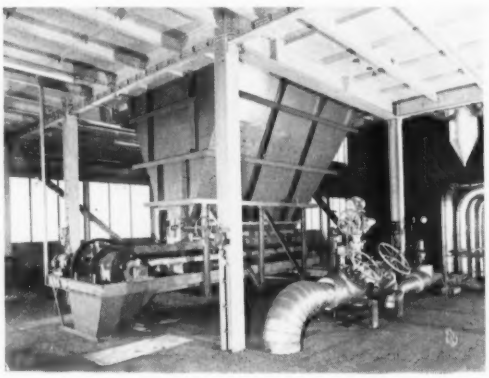
Designed primarily for the use of fines and flotation pyrites, the BASF process has given satisfactory results with a wide range of ores originating from all parts of the world, including coal pyrites and zinc bearing pyrites from Meggen (Germany) which present special roasting problems. Pyrrhotite has been used successfully as well as native sulphur ores with a silicious or limestone matrix and even brimstone in lumps or molten state; the latter, however, requires the admixture of inert materials—such as sand, ashes or clinkers—to permit the formation of a turbulent layer.

In addition to spent oxide which can be used alone or mixed with pyrites, waste materials such as copperas (the waste product of titania manufacture or iron pickling) and sludge acid, can be made to yield their sulphur content, provided that an additional source of heat in the form of a sulphurous raw material or carbonaceous fuel is also present.

In its application to metallic ores the BASF process has been mainly confined to pilot trials which served as a basis for further development work; zinc and copper ores, copper matte, etc., have been roasted successfully and it has also proved applicable for "chlorinating roasting" of pyrites residues, although its suitability for this specialised application has not been finally proved.

Physical Properties of Raw Materials

Size: The BASF process is applicable to a wide range of grain sizes. The top limit is determined by the structure of the raw material and a wide size spectrum is considered to be advantageous. To fluidise a roasting charge with a uniform grain size of 5-6mm. (0.2-0.25 inches) the gas velocities required would be so high that the time of contact between roasting charge and air would be too short to yield a product gas of satisfactory concentration. Friable pyrites up to 10mm. (0.4 inches) can be satisfactorily roasted provided that there is a sufficient proportion of fines 0.2-0.3mm. (0.008-



Ore bin

0.012 inches); such an ore mixture is not unusual when ore preparation entails crushing or coarse milling.

For most pyrites the upper allowable limit of grain size is 6mm. (0.25 inches) while the less friable pyrrhotites and Meggen pyrites have to be crushed to 4mm. (0.17 inches). An admixture of 20-30% flotation pyrites to coarser fines ore is sometimes advantageous.

Flotation pyrites smaller than 0.2mm. (0.008 inches) can be roasted in a BASF roaster at surprisingly high gas velocities and correspondingly high throughput of about 16

tons per m^2 per day, which is equal to about 300kg. sulphur per m^2 per hour. Grain size tends to be increased by agglomeration, a feature which can be promoted by special measures. In consequence, even though the pyrites charge may be of very fine grain size —often predominantly less than 0.05mm.—it is still possible to maintain a stable turbulent layer. In the case of flotation ore the bulk of the cinder is carried onwards in the gas stream and any unburnt or partly burnt ore particles are subsequently roasted in the space above the turbulent layer. This causes the temperatures in the upper part of the furnace to rise by 50°-150°C and has the merit of inhibiting the formation of SO_3 in the roaster gas.

Agglomeration of residues is especially important in the roasting of zinc blende flotation concentrates which takes place at temperatures over 1,000°C. The resultant formation of a relatively coarse calcine permits immediate smelting.

Moisture: Although it is desirable that the furnace charge of flotation pyrites should be as dry as possible the stringent drying conditions of flash roasting are not necessary. The addition of water to the turbulent layer as a means of temperature control is undesirable, but with flotation ore a moisture content of 4-6% promotes a stable bed and ensures longer residence of ore particles in the fluidised zone, so that post-combustion above the fluidised bed is minimised. While as a rule moisture is kept between 2-8%, spent oxide containing up to 14% moisture may be satisfactorily roasted.

Heat Recovery: The utilisation of heat in the BASF plant and process is of technical interest and economic value. Roasting one ton of typical pyrites—containing 45-50% sulphur—produces about 1½ tons saturated steam at 40 atmospheres or the equivalent at a higher pressure or in superheated form. When steam is used for electricity generation by means of a condensing turbine, about 15% of the output meets requirements of the acid plant leaving 85% as credit. Of the total amount of steam generated 50-60% originates from the cooling elements in the turbulent layer and the remainder from the waste heat boiler.

Operating conditions of the steam generator may be varied within wide limits. The BASF system uses forced circulation and steam vessels at 40 and 65 atmos. When integrated with a works power plant, a "once-through" boiler operating at 125 atmospheres is fed. The high pressure steam is superheated to 500°C in the waste heat boiler and then used to generate power.

The life of the cooling pipes built into the turbulent layer is surprisingly long and standard pipes may last more than three years. Pipe fracture in the superheater section of the high-pressure boiler caused by materials failure are readily dealt with as these pipes are accessible. The waste heat boiler also acts as an excellent dust eliminator. Small deposits of dust on the waste heat boiler pipes may be removed by hand or electric shakers.

Mechanical ore feed device.

Specific Capacity

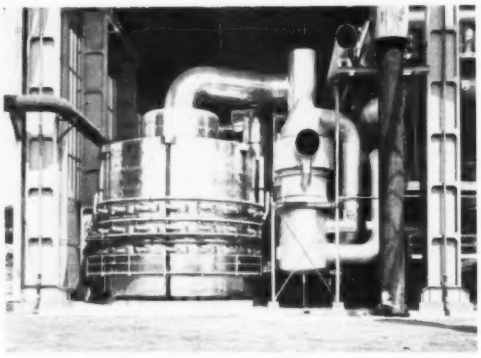
One of the outstanding features of the BASF roaster is the high load factor. Daily furnace charges of 20 tons fines pyrites or 16 tons flotation pyrites per m^2 of roasting surface are commonplace, and by comparison with conventional furnaces, this represents a specific capacity one hundred times greater than in

multihearth furnaces or kilns and ten times greater than in flash roasters. In consequence relatively small furnaces have high capacities.

Normal sized BASF roasters with a daily capacity of 50 tons or 100 tons pyrites containing 45-50% sulphur have a roasting cross-section 2.5 or 5 sq. metres respectively and a throat diameter entry of the furnace charge of 1.8m or 2.5m respectively. Units with daily capacitites of 125 tons pyrites have been operating successfully for about five years and in April 1957 fluidised bed roasters with daily capacity of up to 200 tons pyrites entered production and have continued to operate satisfactorily.

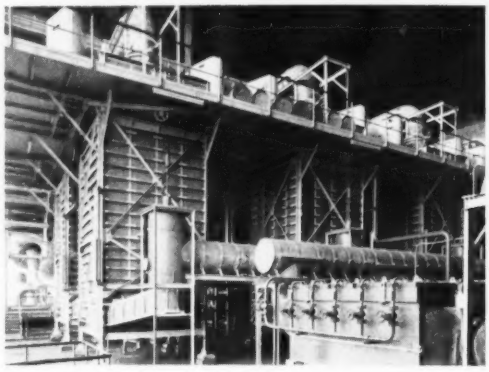
High SO_2 Gas Concentration

As indicated above, roaster gas from turbulent layer furnaces contains normally 12-14% SO_2. The gas is almost as concentrated as that generated by brimstone burners and the low SO_3 content (under 1%) is an added advantage. This high concentration permits substantial economies in the size of the various



K.L.M. contact installation.

units of the gas cleaning section (cyclone, electrostatic precipitator, cooler, wet purifier, dryer and blower) in contrast to equipment required on conventional pyrites furnaces which are usually based on 7% SO_2 concentration. Moreover the BASF has specially developed a five-pass converter to treat roaster gas from turbulent layer furnaces which permits the direct conversion by the contact process of cold cleaned gas containing more than 9% SO_2. It has only one intermediate heat exchanger between the first and second stage while the remaining stages are cooled first by colder roaster gas and finally by cold dry air. This method of cooling has the added advantage of raising the oxygen concentration in the final stages of conversion resulting in greater efficiency. The even supply of cooling gas



Horizontal coolers. Wet E.G.R. chambers made of lead and plastics.

across the entire cross-section of this converter is assured by a large number of mixing jets. Most of these converters were designed for 150 tons per day monohydrate. They have been proved by several years of satisfactory operation, with overall conversion efficiencies of 98-98½% using BASF vanadium catalyst. This type of furnace is remarkably flexible and throughput can be raised to 130% and reduced to 25% of rated capacity and automatic temperature controlled cold air supply to the final conversion stages compensates variations in SO_2 concentration of 6%-10% SO_2.

Residues

Turbulent layer roasting of fine grained pyrites gives rise to dust formation, which in volume exceeds that generated in multihearth furnaces, but is not as great as flash roasting. Turbulent layer roasting of pyrites fines up to 6mm. results in 50-80% of the original volume arising as residues dust. The actual figure is determined by ore structure—e.g. pyrrhotites give little dust, while Kalavassos pyrites produces much dust. Long residence of the charge i.e. a high turbulent layer, increases dust formation. Flotation pyrites give 100% dust but a large proportion is granular, which is attributable to agglomeration in a turbulent layer which one can promote. It has been found that the boiler does not appear to suffer mechanically from the bombardment by the residue dust. Moreover, the dust contained in the roaster gas enhances the heat transfer in

the waste heat boiler. Scaling, if any, is attributable to faulty operation. Earlier difficulties, as for example the formation of deposits on electro-filters have been eliminated.

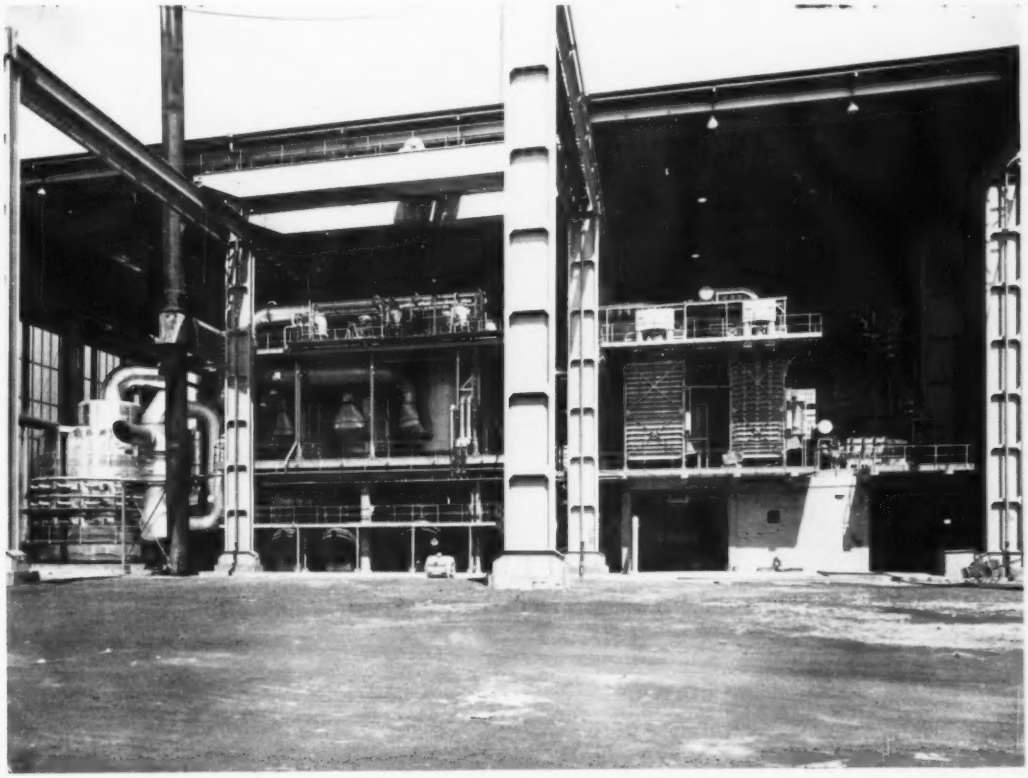
Metallurgy

In the evaluation of ore roasting plant and process the efficient utilisation of sulphur values is no less important than the quality and composition of residue. When roasting non-ferrous sulphide ores the residue is of course the primary material and the sulphur a by-product; the complex metallurgical considerations governed by the application of fluidised bed roasting notably to lead, copper and zinc ores are, however, outside the scope of this review. In view of the importance of the residues five types of pyrites are considered with respect to their behaviour in fluidised roasters:—

(1) Practically pure iron pyrites (e.g. Maremma) and flotation concentrates after extraction of copper and arsenic (e.g. Rio Tinto, Bolidens) : After complete roasting these residues can be used as blast furnace charge, either as fines or agglomerated by sintering.

(2) Zinc pyrites (Meggen pyrites containing 12% zinc sulphide and some Japanese ores) : These may be dead burnt or if part roasted (3% residual sulphur) they are suitable for iron making after the zinc content has been leached.

(3) Pyrites containing substantial amounts of non-ferrous metals, notably copper (e.g. Kalavassos, Bor, Outokumpu, Cuban, Peña, Cyprus Mines Corporation) : When roasting cupreous pyrites in a fluidised bed temperatures of 750-800°C must be maintained, in order not to prejudice subsequent chlorinating roasting and the leaching of minor metals. For this purpose the presence of a small amount of residual sulphur is desirable. As a coarser grain size facilitates leaching, fine ores are roasted for short periods in a low turbu-



General view of the twin fluidised bed roaster each with a daily capacity of 250 tons 100% H_2SO_4.

lent layer to avoid excessive reduction of grain size.

(4) Arsenical pyrites (e.g. Kassandra). When roasting this type of pyrite the bulk of arsenic remains in the residue. In view of the deleterious effect of arsenic on iron, its presence in the blast furnace charge is avoided. If residues contain arsenic only and no other non-ferrous metals, the admixture of crude pyrite to the sinter feed promotes the volatilisation of arsenic.

(5) Pyrites containing copper and arsenic (e.g. Rio Tinto, Tharsis and most other Spanish crude pyrite): Fluidised roasting of these pyrites yields residues which have not so far proved amenable to either a metallurgical or a leaching process, which would permit the manufacture of purple ore free from arsenic or copper. In view of the high qualitative and quantitative needs of the iron industry and because this type of residue cannot provide blast furnace feed unless mixed with substantial amounts of arsenic and copper free ores, the BASF fluidised bed roasting process in its present form is not recommended for this type.

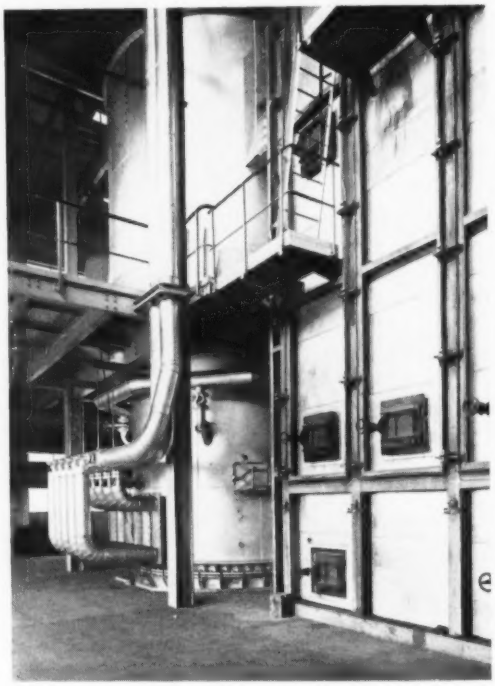
To overcome this situation BASF have developed a two-stage roasting process. At first the arsenical, copper containing, pyrite is pre-roasted in a turbulent layer under conditions of air deficit and indirect cooling. At this stage the polysulphide sulphur is burnt off, resulting in 20% SO_2 roaster gas and part-roasted product charge still containing the bulk of the sulphur content. This intermediate product is recovered from the fluidised bed or from mechanical dust precipitator in the form of part-roasted product. This contains enough sulphur to sustain the reaction in a second fluidised roaster which will yield a virtually arsenic-free residue. Pre-roasting may also be effected in a kiln or multi-hearth furnace.

The bulk of the heat which arises in the transition of FeS_2—Fe_2O_3 is recoverable as steam in the customary manner and the sensible heat of the intermediate product of the pre-roasting stage is also recovered.

BASF have proved the two-stage roasting process in a 100 tons per day pilot plant; a commercial unit of 130 tons per day arsenical pyrites is under construction.

Application

Since 1951 75 BASF turbulent layer roasting units have been built with capacities of



Gas transfer from fluidised bed roaster.

25-250 tons per day (100% H_2SO_4). Thirty further plants are under construction or at the planning stage. In addition to many installations in Germany, BASF plants are installed in the United Kingdom, France, Belgium, Holland, Norway, Sweden, Finland, Spain, Switzerland, Italy, Czechoslovakia, Jugoslavia, Australia, Japan, Union of South Africa, Brazil, and Venezeula. The total capacity of plants installed on 30th November 1957 was 3.3 million tons per annum (100% H_2SO_4) representing about 15% of world acid capacity based on sulphide ores. In the majority of installations the exothermic heat is used in generation of high pressure steam often superheated of 40, 65 or 125 atmospheres. It is estimated that at existing plants and those under construction steam raising capacity amounts to 3.7 million tons per annum. One of the most impressive plants is the recently completed installation at

BASF's own works which, consisting of two equal and independent systems, has a daily capacity of 500 tons (100% H_2SO_4). Each turbulent layer roaster has a roasting cross-section of ten square miles.

Plant Makers

In Germany the manufacture and sale of the BASF turbulent layer roasters is carried out by Messrs. Lurgi Gesellschaft für Chemie und Hüttenwesen m.b.H., Frankfurt a. Main, and in the United Kingdom by Messrs. Simon-Carves Limited, of Cheadle Heath, Cheshire. Also representing the process in Germany is Dipl. Ing. Georg Raschka, of Heidelberg. At most German plants the waste heat boilers are the La Mont type built by Oschatz.

Conclusion

The BASF turbulent layer process which was developed especially for pyrites has many advantages over conventional installations: capacity in relation to size, high concentration of SO_2 roaster gas, accurate control, ease of automation, but above all a high degree of heat recovery of the highly exothermic pyrites roasting process. Steam generation on this scale whether used as such or in the form of electricity represents a valuable by-product comparable with steam recovered in brimstone burning. A further advantage of the BASF turbulent layer roaster is its versatility in that in addition to sulphide ores it can effectively burn elemental sulphur and native sulphur ore. In recent years a number of low grade sulphur ore deposits have been developed and turbulent layer roasting provides an economic means of using this source of sulphur supply. The production of high quality, low sulphurous, non-arsenical, non-cupreous residues is a significant factor in the economics of pyrites use. The difficulties of producing an acceptable iron residue from crude arsenical, copper containing pyrites—the most abundant types in Spain and Portugal—is therefore a handicap which the development of a two-stage roasting process may help to resolve. Comparative roasting costs are, however, a major factor under the highly competitive conditions in supply of sulphurous raw materials. In the past three years the application of fluidised bed roasting — and in particular the use of BASF process and plants — has made a decisive contribution to the improvement in the economics of pyrites use in acid manufacture especially in Germany. In doing so it was an important factor in preventing brimstone making even greater inroads in Europe's sulphur markets than have already occurred.

We are indebted to Badische Anilin-& Soda-Fabrik of Ludwigshafen for information and illustrations placed at our disposal and acknowledge articles published in Chemie-Ingenieur-Tecknik 24 104(1952) and 29 563(1957) by Dr. rer. nat. Adolf Johannsen and Dr. phil. nat. Willi Danz.

SULPHUR FILTRATION

The Niagara Horizontal and Vertical Filters

THE presence of impurities in brimstone impairs the efficiency of sulphuric acid manufacture. Burners get clogged and ash accumulates in the combustion chamber and above all impurities carried in the SO_2 gas stream affect contact acid plants by fouling the catalyst bed and reducing its rate of conversion.

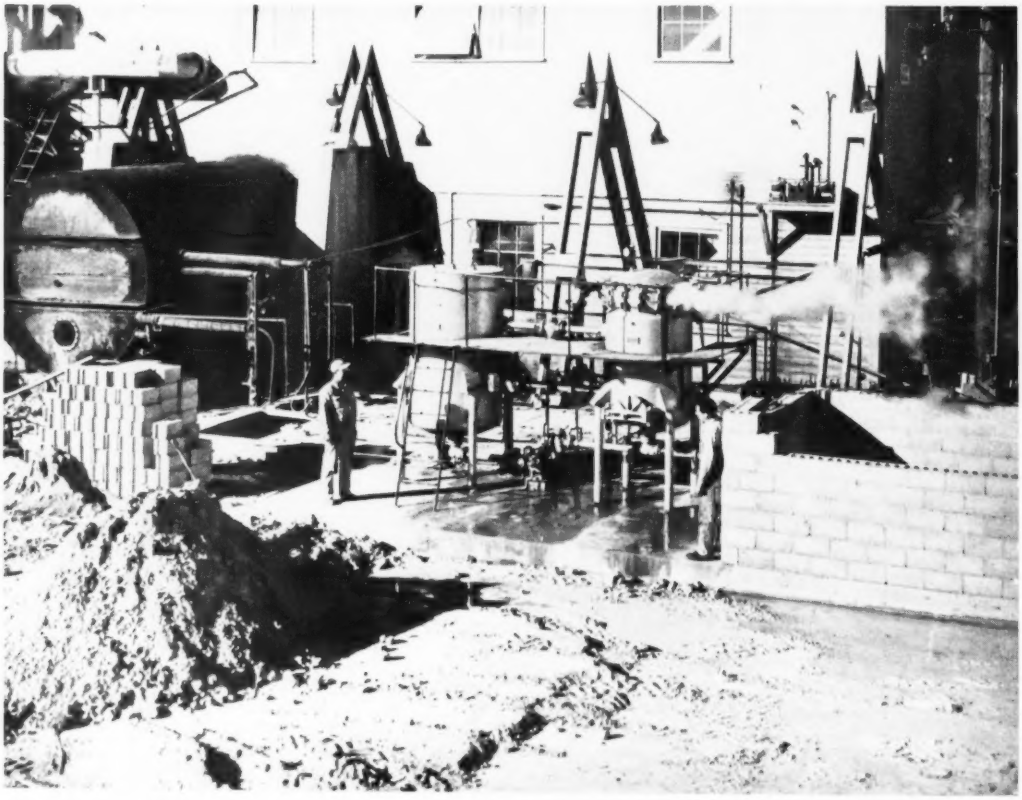
Impurities may be in the form of either ash or hydrocarbons. Freshly mined U.S. Frasch bright sulphur contains about 0.03% to 0.01% ash, while dark Frasch sulphur has about the same ash content in addition to a small amount of carbonaceous materials. The transportation of sulphur by sea or land and its inevitable exposure to atmosphere leads to contamination by dirt, air borne chemicals and solids and by the time it is used the ash content has generally increased to about 0.10% and at times even higher.

The commonest means of removing the ash and other solids is by settling. Large sunken pits are used and the molten sulphur is allowed to stand for 24 hours or more to permit the solids to settle. The coarse particles settle rapidly but the fine impurities settle slowly and after 24 hours 0.02% ash or sometimes more remains. To protect the catalyst bed of the contact sulphuric acid plants gas filters ahead of the convertors have been tried but their effectiveness varies appreciably.

The presence of hydrocarbons in native sulphur deposits is widespread particularly in

the case of the dome deposits around the Gulf of Mexico. Hydrocarbons in sulphur used in the manufacture of sulphuric acid by the contact process is undesirable; they are not removable by means of settling, and only filtration at times combined with special treatment, is capable of doing so to any extent.

The research activities of the Niagara Filter Corporation revealed the advantages of filtration of molten sulphur compared with the use of settling pits and gas filters. In 1947, following four years extensive research, the company's first commercial installation was placed in operation in the United States. Since then the company has installed more than sixty plants in the United States and also at many important European brimstone acid plants.



General View of a Niagara filtration plant with a daily throughput capacity of 100 tons dark sulphur.

Construction of Niagara Sulphur Filters

The Niagara molten sulphur filters are manufactured in a wide range of sizes to cover the requirements of plants producing from 25 tons to 1,000 tons of sulphuric acid per day. They are constructed in two types, vertical and horizontal tank units, depending on the type of filter housing. Both contain a series of vertical double faced screen assemblies—filter leaves—consisting of three layers of stainless steel wire mesh. The outer leaves are fine meshed and the centre piece is coarse; they are tightly bound by riveting into a tubular one-piece welded frame. Delivery of sulphur to the manifold is through a machined nozzle welded to the bottom of the frame.

Vertical: This type has an hydraulic lift mechanism to raise the cover and swing it aside exposing the entire bank of leaves. At the side of the filter tank there is a clean-out door through which the filter cake may be removed. The cover is operated by handwheels and bolts or a special quick-opening mechanism.

Horizontal: In this type the filter leaves are mounted on a retractable carriage which rolls out on a monorail and is operated either by hand or hydraulically.

The vertical as well as the horizontal types have all-welded carbon steel filter-tanks enclosed in steam jackets designed for pressures of 50lbs. p.s.i. or higher. To counteract the corrosive effect of sulphuric acid formed by the oxidation of sulphur, the fine filter wire is made of molybdenum stainless steel.

Operation

Since the fine wire cloth is not generally in itself satisfactory, a precoat layer of diatomaceous earth is established on the filter leaves to act as a filter medium. The precoat layer is formed by pumping a slurry of filtered sulphur and diatomeaceous-earth from precoat tank to filter until a clear sulphur is obtained. Following this operation, which takes about 10 to 15 minutes, actual filtration begins. Unfiltered sulphur is pumped from the melting pit to the filters, until the final pressure of 65lbs. p.s.i. has been reached. With bright sulphur it is possible to maintain the filtration cycle for 168 hours, without any interruption for cleaning.

With sulphur containing a high percentage of solids of granular nature, for instance crude Italian sulphur and sulphur flotation concentrates, the need for filter aids is eliminated. In the filtration of such sulphur materials the molten sulphur is recirculated through the filter and back to the melting pit to establish a cake which then will act as the filtering medium. The filtration can usually be continued for only 4 hours by when the cake has built up to 1 to 1½ inches.

Cleaning

At the end of the filtration cycle, when pressure has risen to 65lbs. p.s.i., the filter is shut down for cleaning and any liquid sulphur remaining in the filter tank is drained back to the melting pit. With vertical filters the cover is lifted and swung aside, to allow the cake on the top part of the leaves to cool and solidify becoming brittle. The cake is fractured by tapping the top of the leaves with a rubber mallet and after falling through into the filter tank it can be removed through the cleanout door on the side of the tank. The cleaning of the horizontal type filters is simpler since the carriage containing the whole set of filter leaves can be rolled out. The cake is removed in the same way as with the vertical type and it falls down into a hopper. This type permits resumption of operation within one half-hour.

Filtration of Bright Sulphur

Once the filter leaves are properly precoated with diatomaceous earth bright sulphur is the easiest quality to filter. As indicated above, filtration can be carried on for as long as 168 hours though the usual filter cycle is 3 or 4 days. Unfiltered sulphur should be neutralized with hydrated lime to limit corrosion. The filtered sulphur contains 0.003% ash or less. For this type of sulphur vertical filters are commonly used as the quicker cleaning of the horizontal does not offer great advantages.

Filtration of Dark Sulphur

As this type of sulphur contains hydrocarbons, which form a thin impervious layer on the precoat thus reducing flow rate and cycle period, the admixture of diatomaceous earth is necessary. Some types of dark sulphur contain hydrocarbons which will not be retained by the diatomaceous earth filter cake and these entail a special treatment, usually with bleaching clay. In some cases chemical treatment is necessary to reduce the hydrocarbons or to make them amenable to filtering. Vertical as well as horizontal filters are used, but as the filter cycle will not normally be longer than 12 hours horizontal filters are preferred on account of greater ease of cleaning.

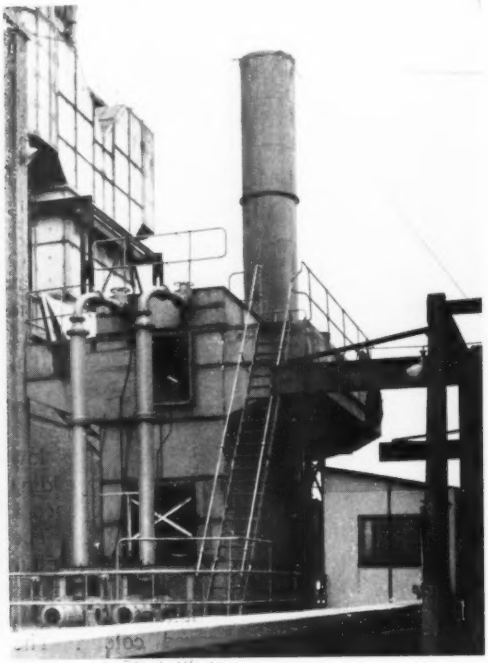
Filtration of Sulphur Concentrates

In recent years the beneficiation of low grade native sulphur ores by flotation yielding a concentrate of 85-95% sulphur has opened a new field of application to filtering. The horizontal filter handles the flotation sulphur concentrate and when all insoluble impurities have been removed the resultant product is 99.5% pure and has improved colour. The filtration yield will be about 90%-95% depending on the amount of solids to be removed and on the residual sulphur contents of the filter cake usually about 60%-70%. Sulphur thus filtered will have an ash content of 0.10% to 0.15% and bitumen, if originally present, will be reduced.

We are indebted to Messrs. Niagara Filters, Europe, for information and illustration placed at our disposal.

SULPHUR RECOVERY FROM FLUE GAS

DURING the past 25 years, much experimental work has been carried out at power stations in the London area with the object of removing sulphur oxides from the chimney gases. The two principal processes developed in this work proved costly to operate and since no useful products were recovered, there was considerable scope and incentive for the development of a process which would enable useful products to be recovered for sale. With these disadvantages in mind, the Metropolitan Borough of Fulham approached Simon-Carves in 1938 and, as a result of this co-operation, the Fulham-Simon-Carves process was evolved, which produces saleable ammonium sulphate and elemental sulphur. This work was held up due to the war and later due to rigid control of capital expenditure immediately after the war. Experiments to assess the process were carried out at the Fuel Research Station*, and more recently a pilot plant has been installed at North Wilford Power Station, Nottingham; this plant is on such a scale that it will allow a commercial appraisal of the process to be made.



General view of the flue gas washing plant.

The North Wilford plant has been designed to handle the gases produced by the combustion of approximately 7 tons of coal per hour (56,000 c.f.m.), the sulphur content of the coal being in the range 0.5 to 3.0%. The gases are scrubbed in a closed cyclic system with a solution of ammonium salts, to which is added the appropriate amount of ammonia to react with the sulphur oxides absorbed from the flue gases. The ammonia is used in the form of concentrated ammonia liquor drawn from local gas works. Inter-action of the sulphur oxides with the circulating liquor produces a solution containing ammonium sulphate, ammonium sulphite, ammonium bisulphite and ammonium thiosulphate, the latter compound being produced mainly because of the ammonium sulphide content of the gas works liquor. Water and ammonia are added as necessary to maintain a constant concentration and composition of the scrubber liquor, whilst the quantity in circulation is maintained at a constant value by the withdrawal of liquor for working up into the final products.

The initial objective of the process was the production of ammonium sulphate. It was therefore necessary to oxidise the various other sulphur compounds which were also present in the liquor. This proved to be a matter of some difficulty owing to the presence of the ammonium thiosulphate. The method finally adopted consisted in heating the liquor under pressure with a small proportion of sulphuric acid; by this means, the ammonium sulphite, bisulphite and thiosulphate react together to produce ammonium sulphate and elemental sulphur. The sulphur is discharged from the autoclave in the molten state, whilst the ammonium sulphate remains in solution and requires crystallisation by conventional processes.

Since the experimental work at North Wilford is still in its early stages, it is difficult to quote figures for production owing to the

* See Quarterly Bulletin No 13, page 22

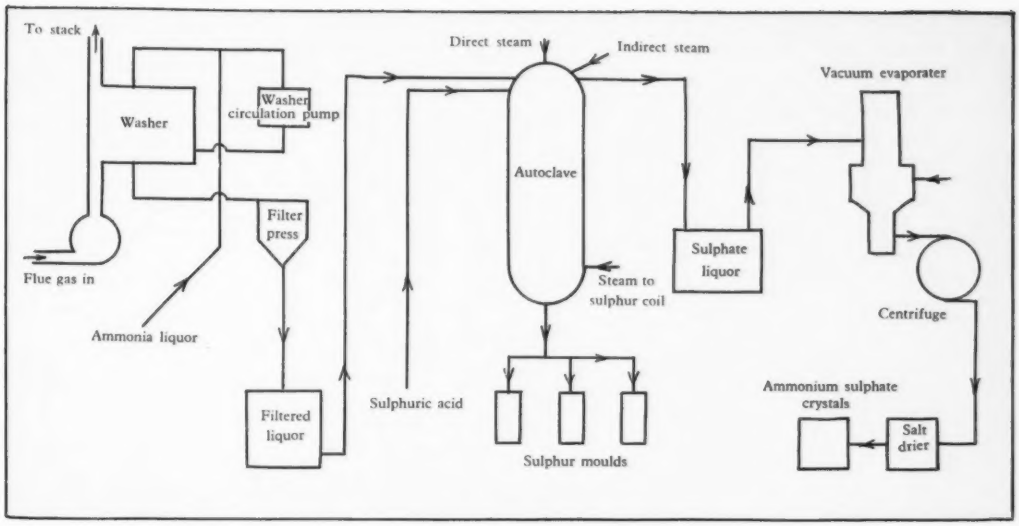


Fig. 2

intermittent operation of the plant. During the four months in which the plant has so far operated, approximately 110 tons of ammonium sulphate and 20 tons of sulphur have been produced. Experience has shown in this period that it is possible to produce ammonium sulphate conforming to the Grade I Specification of the British Sulphate of Ammonia Federation; the sulphur produced is also of a high degree of purity.

We are indebted to the East Midlands Division of the C.E.A. for the account and illustrations placed at our disposal. The photograph is Crown copyright.

MECHANICAL OXIDE DISCHARGER

A NOVEL machine, which will overcome many of the difficulties at present associated with oxide purification and at the same time facilitate the handling of oxide, has been patented by Newton Chambers & Co. Ltd., and is being made at their Thorncliffe works near Sheffield. It is a mechanical oxide discharger which can be built into any system of oxide handling plant, whether simple or comprehensive. The first one is already in successful operation in conjunction with the tower purifiers built by Newton Chambers on the National Coal Board Coking Plant at Monkton, Co. Durham. Others have been ordered for the Isle of Grain tower purifiers of the South Eastern Gas Board, and the Bradford Road towers of the North Western Gas Board.

In operation, the mechanical oxide discharger eliminates a laborious and unpleasant task previously done manually, and contributes to the smooth, economical operation of tower purifiers. The removal of spent or partially-spent oxide from purification plants has always been a problem. On the earlier type tower purifier installations men with picks and shovels have had to enter and clear the containers, and the work has been unpleasant as well as slow, and consequently costly.

The new Newton Chambers machine empties the containers by mechanical means. In brief, a series of steel curved ploughs, operating on a revolving turntable, cuts and propels the fouled oxide towards a central opening in the container, down which it is discharged. The machine is designed for use in conjunction with circular containers free from internal obstructions such as the radial bars supporting the grids which occur in the earlier types of containers. The important feature of the containers is the removable centre tube. When in position in a tower, the centre tube, of course, conveys the gas either into or away from the tower. Its

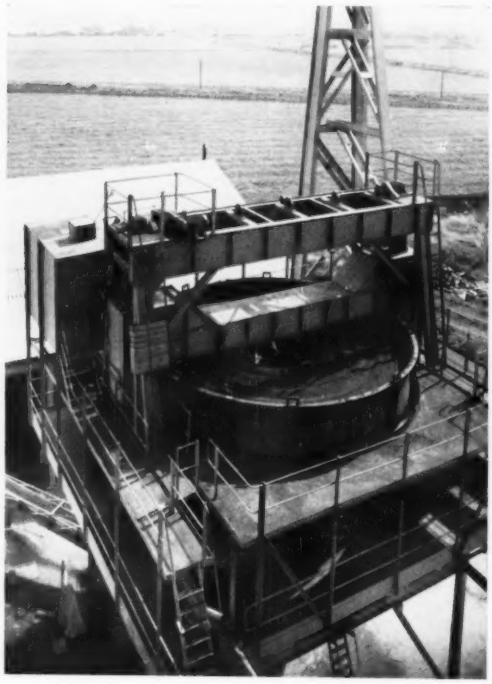
removal provides a central opening within the oxide mass. The machine consists essentially of an elevated turntable which rotates the container against a fixed cutting mechanism

Spent oxide container being lifted from the stocking tower for transfer to the rotating turntable on left.

carried on a gantry. Means are provided for moving the gantry and the cutting mechanism away from the turntable along guides so that the container can be lowered on to the machine. The sequence of events during the emptying of a container is as follows. First, the container is removed from the stocking tower, where it is stored, by means of the overhead crane and lifting frame, and is lowered on to the turntable, where it is located in position. Secondly, the centre tube is removed, again using the overhead crane. Thirdly, the gantry carrying the cutter beam is moved, either manually or mechanically, into position over the container, and is secured there.

The turntable is then set in motion and the cutter beam made to descend at a constant speed so that the total depth of oxide is discharged in a time which can vary between 25 and 45 minutes, to suit the capacity of the ancillary oxide-handling plant. The descent of the cutter beam is stopped automatically immediately above the grid level. The cutting mechanism consists of a series of ploughs attached to the underside of the cutter beam. As the turntable rotates, the oxide is cut and propelled by the curved ploughs towards the opening left on removal of the central tube. In this way, a steady and continuous discharge of oxide can be collected below the container, whatever the original condition of the oxide.

When the cutting of the oxide has been completed the cutting beam is lifted, the speed of raising being considerably faster than the



Spent oxide box on rotating turntable being cleared by plough.

speed of descent, and it is removed from its position above the container. The container is finally cleaned and swept free from oxide and is transferred to the filling turntable, where it is re-charged with oxide and then transferred to the stocking frame ready for use in a working tower.

We are indebted to Messrs. Newton Chambers & Co. Ltd., for information and photographs placed at our disposal.

By-Product Sulphuric Acid from Up-Draught Lead Sintering

THE plant development which was completed by Broken Hill Associated Smelters Proprietary Ltd., at Port Pirie, South Australia, early last year, whereby lead smelter gases were made to yield their sulphur content for manufacture of sulphuric acid, represents an important extension of the field of sulphur recovery from industrial gases. The key to the development, which was initiated in 1947, is the modification of the standard down-draught Dwight Lloyd sintering machines to an up-draught system which removes the major basic disability of excessive lead precipitation in wind boxes necessitating frequent cleaning and interruption of gas delivery. It also overcame purely local difficulties which were principally (a) the need to avoid reducing the production capacity of the lead sinter and (b) the prevailing high temperature of cooling water required an abnormally high SO_2 concentration to allow the customary output of 98% acid.

Up to 1954 a contact acid plant built in 1924 and based on the use of SO_2 in zinc smelter gases had been operated by the Company. It was closed because of the removal of the zinc smelting operation to Risdon, Tasmania, and development of the new source of sulphur supply led to the decision to instal a new acid plant and to design and build a suitable gas purification train. The new sulphuric acid plant is of conventional design, and after its completion in October, 1956, it was temporarily fed from a brimstone burner to produce at 60% of its nominal capacity of 50,000 tons per annum (100% H_2SO_4). On completion of the purification section, supply of sulphurous gas from sinter machines was substituted, and since early 1956 the plant has given outstanding service on this basis.

Sinter Plant

The new up-draught machine is 10ft. by 99ft., some 44ft. longer than the old down-draught installation. It was made to fit in with all the existing ancillary units, and its installation resulted in comparatively small disturbance of normal operations. The features relevant to this review concern mainly the generation and removal of roaster gas.

A constant suction of minus 0.1 to 0.2 inches W.G., just sufficient to remove the gas,

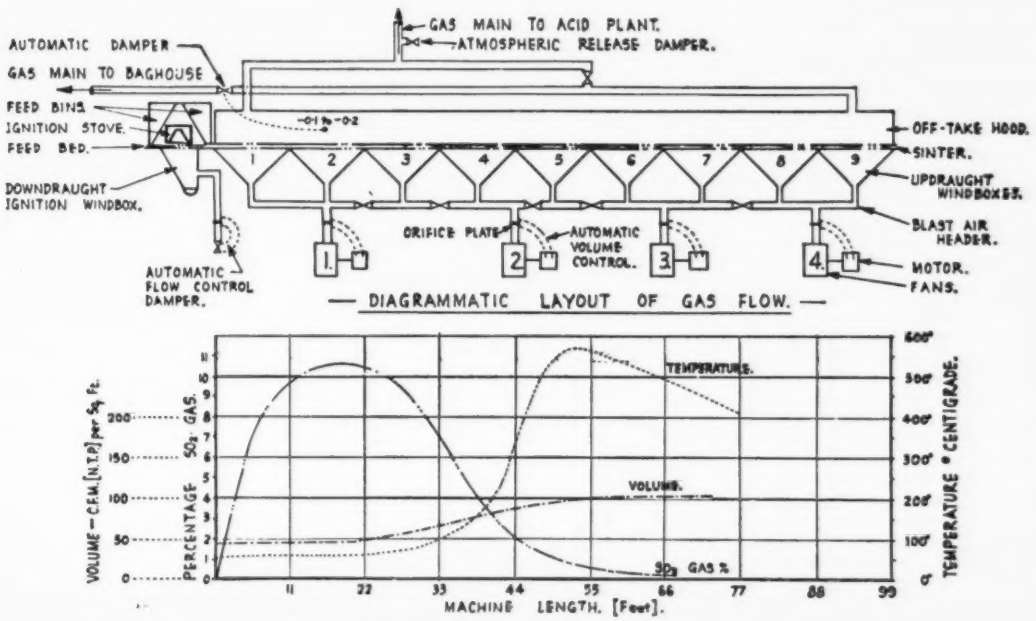


Fig. 3. Gas flow diagram and roast gas SO_2, temperature and volume graph.

is maintained in the gas-collecting hood over the full length of the machine. "Acid plant" gas is drawn off 10ft. from the feed end through a 5ft. diameter off-take flue to the purification gas main. There is a by-pass to the bag-house gas flue, and an excess suction release damper, which trips at minus 1.7 inches W.G. to admit air to the flue, protects the purification section.

General view of sinter unit showing gas off-take hoods.

Gas Collection

The bulk of the sulphur is eliminated from the sintering charge over the first 40ft. of the strand. The residual amount of SO_2 is carried along with the tail or bag-house gases, which are taken off at the discharge end. New sintering charge contains an average of 9.35% sulphur; as this is mixed with crushed returns in the proportion of 55:45 the following machine condition prevails:—

Sulphur in feed	6.8%
Sulphur in final sinter	1.8%
Sulphur in crushed returns	3.2%

Sinter gas which contains 8-9% SO_2 is determined by the blower rating set at the acid plant, and is independent of the sintering operations which normally yield 60 tons sinter per hour. At N.T.P. the gas volume is 19,500 cubic ft. per minute and the temperature of the gas delivered to the purification train is kept at 180° to 220°C. by controlling the volume drawn from the feed and if necessary by water spray cooling in the hood 30 to 50ft. from the feed end.

Recovery

Although at present only about 75% of sulphur content of the sinter gas enters the purification section it has been proved that 85% of the eliminated sulphur can be collected at acid plant grade by increasing the off-take volume. This cannot however be implemented at Port Pirie as the volumetric capacity of the converter is limited to 19,000 cu. ft. per minute on one blower. The maximum possible degree of recovery is still being investigated.

Purification Section

The gas purification section, as is the case at most metal smelters, is extensive and is designed not only to remove impurities to make the gas suitable for conversion into sulphuric acid but also to recover those impurities which are of economic value, especially lead and cadmium.

The purification section is designed to handle up to 22,000 cu. ft. of sinter gas per minute at normal temperature and pressure (0°C. and 760 m.m. pressure) and is divided into two parts connected by an overhead lead gas main some 600ft. long. The first part, situated adjacent to the sinter plant, removes most of the solid impurities from the gas and also reduces its temperature so that it may be passed, without trouble, through the 200ft. long connecting main to the final purification and conversion sections.

The purification section is sub-divided into primary and final sections :

Primary : Gas from the sintering plant is delivered by a Richardson paddle-wheel- type fan to the top of the conditioning tower 33ft. high and 24ft. in diameter. The gases enter it at the top on one side, descend and then ascend on the other side, finally leaving the tower through two 5ft. diameter lead-lined steel flues and pass to the dry cottrell.

The gas is cooled from 200°C. to 110°C.-120°C. by fresh water admitted to the inlet side of the tower through atomizing sprays,

the number of sprays in operation being controlled automatically from the thermostats fitted into the entrance of the dry cottrell. The gas is conditioned to render it conductive and therefore capable of precipitation in the dry cottrell by the addition of SO_3 gas, formed in a small converter adjacent to the tower from pure SO_2 gas supplied under pressure from the main blowers at the conversion section. About 40-60 cu. ft. of 6-7 per cent SO_3 gas per minute is admitted to the tower near the bottom on the outlet side. The aim, of course, is to evaporate all the water into the gas stream, but in practice a small run-off occurs when all of the twenty-three sprays are working, due, probably, to impingement on the walls, etc., and this is drained to a lead-lined concrete sump 14½ft. wide and 4½ft. deep from which it is pumped periodically to the dry cottrell sump. The sump at the conditioning tower also serves to collect the material washed out of the tower during the weekly flue cleaning.

The conditioned gas at a temperature of 110°C.-120°C. then enters the Lodge-Cottrell dust precipitator in which the bulk of the solid material in the gas is removed. The precipitator is divided into two parallel chambers each consisting of three compartments in series, and each chamber can be isolated for repairs by means of drop dampers and, if necessary, blank plates in the inlet and outlet flues.

The first of two sets of mist precipitators known as the wet cottrells, are designed to remove practically all the remaining solids in the gas as well as the bulk of the acid mist contained in it. The gas leaving this unit normally shows only a faint trace of mist when viewed through the outlet main from one end with a light at the other end.

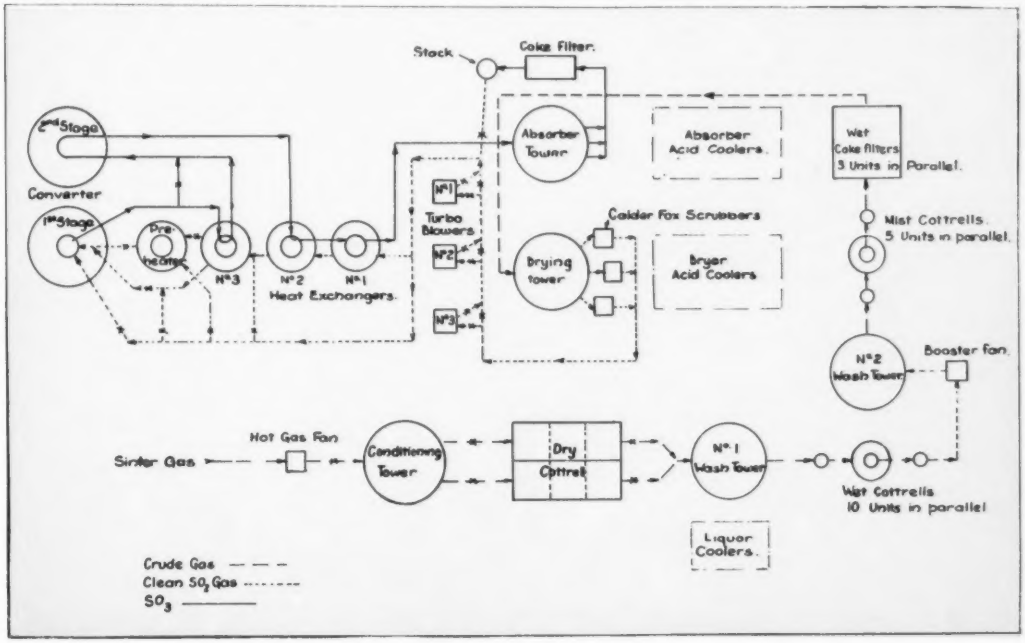


Fig. 4. Gas flow diagram of the sinter gas.

The first wash tower serves to cool, humidify and scrub the gas leaving the dry precipitator before it enters the first set of wet precipitators. Two horizontal LaBour pumps maintain a circulation of about 36,000 gal./hr. of slightly acidulated water over the tower, and this is distributed over the top of the packing by 32 sprays, each consisting of an inverted lead saucer supported beneath a nozzle.

About 3,000 gal. of fresh water is fed into the circuit per hour, and a corresponding amount of "foul" liquor bled off into the wet cottrell settling sump and thence to waste. In this way the concentration of arsenic in the

circuit is held at an average of 15 parts per million.

The circulating liquor is cooled in five lead-lined steel tanks each 30ft. 6in. long, 6ft. 6in. wide, and 6ft. high, operating in parallel, each containing 36 2in. lead pipe coils with a total heat transfer surface of 2,200 sq. ft. per tank.

Sea-water is pumped through the coils from the works pumping station, the five tanks taking a total of some 30,000 gal./hr. The tanks act as settlers for the solids scrubbed out by the tower, and the tanks are emptied periodically, the sludge draining to the wet cottrell settling sump.

Average temperatures over the wash tower circuit are as follows :

Liquor	
In tower	42°C.
Ex tower	50°C.
Ex coolers	42°C.
Sea-Water	
In coils	20°C.
Ex coils	32°C.
Gas	
In tower	120°C.
Ex tower	42°C.

The sinter gas, now cooled to 40°C.-50°C. and practically free from solid material, passes through a 3ft. 9in. diameter lead main some 600ft. long to the final purification section, where it is cooled to 30°C.-35°C. to ensure that it contains less water than that required to make 98 per cent sulphuric acid, purified to remove traces of solids and acid mist and, finally, dried so that a clean dry gas enters the conversion section.

Final Section : The gas-boosting fan situated at the beginning of the final purification section is provided to reduce the suction on the lead work following it. The chief function of the second wash tower, which is identical with the first except for the packings, is to cool the gas to 30°C.-35°C., by direct scrubbing with sea-water; the system was adopted in view of excessive summer temperatures of fresh-water circulation. This effectively cools the gas, but of course leads to a loss of SO_2 gas. There are also definite indications that severe corrosion of the lead-work in the lower portion of the tower is occurring.

It is anticipated that the gas will be cooled sufficiently in the long overhead main and the

General view of the final purification section for the sinter gas. Left background, the heat exchangers and preheaters.

remainder of the plant during the winter months to enable the tower to be operated "dry," thus reducing the SO_3 loss and corrosion on the lead-work.

In the mist cottrells any mist or fumes still left in the gas is precipitated to make the gases leaving the unit optically clean. There are ten units in parallel, each consisting of a circular steel-framed lead shell containing 37 6in. diameter lead tubes 9ft. long.

The purified gas is dried in a single drying tower 32ft. high and 20ft. diameter, constructed of mild steel with an acid-resisting brick lining set in acid-proof cement. Sulphuric acid at 95 per cent strength is circulated over the tower at the rate of 300 tons/hr., and in descending through the packing removes virtually all of the water contained in the ascending gases, the average moisture and acid mist content being 1 to 1.5 mg. and 0.005 to 0.015 mg. per cu. ft. respectively.

The acid leaving the tower is brought up to 95 per cent strength again by the addition of 98 per cent acid from the absorption circuit and then cooled in a series of 4in. diameter cast-iron pipes, externally irrigated by sea-water, before returning to the circulating tanks to be pumped to the head of the tower again. The total length of pipe for cooling purposes is 3,780ft. The excess acid in the circuit is fed to the absorption circuit for dilution of the absorber acid.

The acid temperatures in normal operation are as follows :—

Exit towers	28°C.
In coolers (after strengthening)	28°C.
After cooling	25°C.

The dried gas leaves the tower through three outlets, each connected to a Calder-Fox scrubber followed by a cast-iron catch pot to remove entrained acid before it enters a 3ft. 6in. diameter mild steel main leading to the blowers and thence to the conversion and absorption sections.

Conversion and Absorption Section

The gas is drawn through the sections already described, with the assistance of the hot gas fan and booster fan, by centrifugal blowers, and delivered to the heat exchangers and converter in which it is heated and the SO_2 converted to SO_3. The gas, cooled in the heat exchangers and an atmospheric cooler, is then delivered to the absorbing tower, where the SO_3 is absorbed in 98 per cent sulphuric acid. The waste gases, chiefly N_2 with small quantities of O_2, SO_2 and SO_3 are finally discharged to atmosphere through a 76ft. stack after passing through a small coke filter to remove entrained acid.

The gas flow through the heat exchangers, pre-heater (if necessary) and converter, is as follows :

The three heat exchangers are arranged in line, and the gas from the blowers enters the space surrounding the tubes of the first exchanger at the bottom, and ascending passes

Sulphuric acid installation; converter and heat exchangers in the centre with temporary brimstone burner (left).

to the top of the second exchanger. It then descends in the second and ascends in the third exchanger, in each case around the tubes. From the third heat exchanger the gas passes, within controlled temperature limits, through the first stage of the converter, where the larger proportion of the SO_2 is converted to SO_3.

The reaction is exothermic and while the increase in the gas temperature as it proceeds through the catalyst accelerates greatly the rate of conversion of SO_2 to SO_3, it also limits the maximum percentage conversion obtainable.

To enable the reaction to proceed at a higher efficiency, the gas flows through the first stage of the converter, then downwards through the tubes of the third heat exchanger, giving up some of its heat to the SO_2 gas which circulates around the tubes. The partly-converted and cooled gas enters the second stage of the converter and in passing over the catalyst in this stage, most of the remaining SO_2 is converted to SO_3. The hot gas leaving the converter then passes through the tubes of the first two heat exchangers in series, being cooled in its passage. It is further cooled in a series of parallel atmospheric cooling pipes before entering the absorber. In addition to the main flow through the heat exchangers, by-pass mains are provided to maintain the catalyst beds within temperature limits favourable to maximum conversion efficiency.

The temperature of both masses in the first stage may be regulated by admitting cold gas from the blowers to the inlet main of the converter and also between the two masses.

The temperature of the gas entering the second stage may be reduced by admitting cold SO_2 to the SO_2 inlet main of the third heat exchanger and increased by by-passing hot gas directly from the first to the second stage of the converter.

For starting up the converter from cold and also for providing additional heat after prolonged stoppages, an oil-fired tubular pre-heater is provided.

The plant is now satisfactorily established and represents an important addition to Australia's programme of enhancing the use of indigenous sulphurous raw materials. Conversion efficiency applied to the 6-8% SO_2 gas is 90-94%. Current output is about 1,100 tons per week, 10% in excess of nominal capacity. Peak production levels of $8\frac{1}{2}$ tons per hour corresponding to 74,000 tons per annum (100% H_2SO_4) have been achieved. Output is in the form of 98% acid. Like all by-product acid operations at metal smelters it shows favourable economics. There is provision in the Sulphur Bounty Act for subsidy in case of need, but it is thought unlikely to be necessary.

We are indebted to Broken Hill Associated Smelters Pty. Ltd., also to the Australasian Institute of Mining and Metallurgy for material made available to us on Up-draught Sintering and Sulphuric Acid Production at Port Pirie from their 'Proceedings' (180), Dec. 1956.

ACID LEACHING OF URANIUM ORES

Gunnar Mines Ltd.

LEACHING and the subsequent recovery from the leach liquor of uranium oxide concentrate permits the economic extraction of the small quantities of finely disseminated uranium oxide from a large volume of gangue material. With its predominant use of dilute sulphuric acid as a leaching agent this young and rapidly expanding industry represents the most recent important consumer of sulphur. The processing of uranium ore, notably in Canada and in the U.S.A., is based on common factors and in most instances it follows practically identical lines. Even in the Union of South Africa where uranium recovery is integrated with gold ore production the leaching and concentration stages are similar. The developments and the flow sheet at the Lake Athabasca plant is therefore thought to provide a representative picture.

The discovery of uranium deposits in 1952 on the shores of Lake Athabasca, Saskatchewan by geologists from Gunnar Gold Mines was rapidly followed by their systematic survey and a preliminary diamond drilling programme. In August, 1953, ore samples from these drillings, which covered the projected site of the company's open pit and some of the

Gunnar Uranium Mines. General view.

underground mines, were forwarded to the Bureau of Mines, Ottawa, Ontario, for evaluation and metallurgical test work.

Following these tests Gunnar Uranium Mines supplied a 50 ton sample, taken from an outcrop in the future open-pit area, to the Bureau of Mines and commissioned them to carry out full tests and make recommendations on process and plant design. Research work established that the acid-sodium chlorate process was the most suited for the dissolution of uranium and that for the recovery of the uranium values from the solution ion exchange with magnesia precipitation was the most suitable.

Plant design of an integrated unit consisting of milling, crushing, leaching and power units and the sulphuric acid plant, located 500ft. north east of the mill building, was now completed and put into operation on the 23rd August, 1955. No operating troubles were encountered and the original milling capacity of 1,250 tons daily was constantly exceeded and in March, 1957, capacity was expanded to 1,650 tons daily.

Description of Operations

Crushing. Fed from an open pit, the gyratory crusher discharges ore with a maximum size of 5 inches. The crushing plant is of standard design and operates in a closed circuit. Equipment includes:

One 36" x 48" Traylor Jaw Crusher.
One 42" Nordberg gyratory crusher
One 5½' Symons Standard Cone Crusher.
One 5½' Symons Shorthead Cone Crusher.
Two 5' x 8' x 1½" Symons rod deck screens.
Two 4' x 8' x 1¼" Symons rod deck screens.

Grinding. Material from the primary grinding is split into two duplicate circuits, classified and transferred to a closed circuit containing a Marcy ball mill where it is ground to 57% minus 200 mesh at 69% solids. Overflow from the classifier at 39% solids is then transferred for thickening prior to leaching.

Each circuit contains the following equipment:
One 9' x 12' Marcy Rod Mill. Operates at 17 R.P.M. load factor 100 amps. 3" rods at 0.97 lbs per ton milled.
One 78" Aikens Classifier.
One 9' x 10' Marcy Ball Mill. Operates at 20 R.P.M. load factor 120 amps. 1½" balls at 0.87 lbs per ton milled.

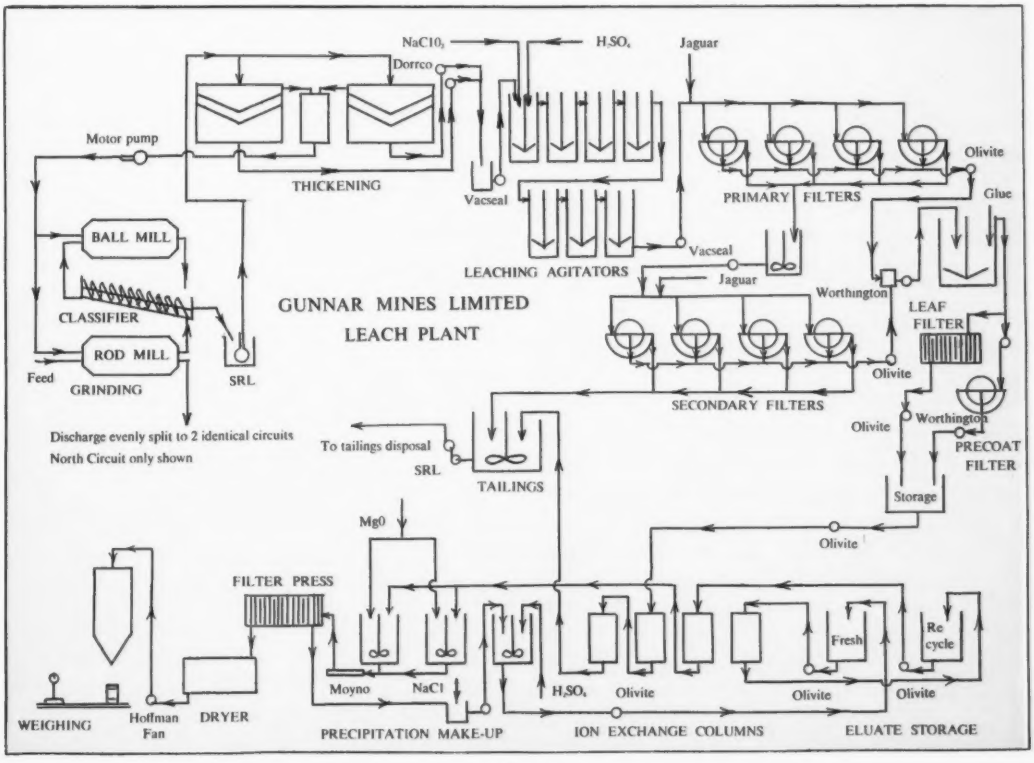


Fig. 5.

Leaching. Overflow from the thickener is returned for grinding and the underflow, thickened to 57-60% solids, is transferred to the leaching agitators, which operate in series. Sulphuric acid and sodium chlorate is introduced into the No. 1 agitator when it becomes exhausted so that pH is maintained at 1.7. This is automatically controlled and recorded in No. 1 unit. Sulphuric acid consumption is 179 lbs. of 93% H_2SO_4 per ton of ore compared with 1.61 lbs. of sodium chlorate which acts as the oxidiser. This chemical reaction raises the heat to 100°F; retention time is 24 hours and the pulp is then ready for filtration.

This plant section consists of:

Two 50′ Dorr thickeners — each with three compartments designed area of 9 sq. ft. per ton of solids daily.

Seven 20′ x 20′ leaching agitators. Wood stave tanks with Dorr AA mechanism of 316 stainless steel, automatic pH controls are fitted to each agitator.

Filtration and Clarification. Double stage filtration is carried out in 11½ft. diameter x 16ft. filters with the aid of Jaguar filtration reagent consumed at a rate of 0.10 lbs. per ton. Filtering is carried on at 840 lbs. per sq. ft. per 24 hours and the cake is repulped with barren solution from ion exchange and discharged to tailings. All filtrate is combined and sent to a clarification thickener the supernatant liquor is clarified to 50 ppm. by a Moore conventional type leaf clarifier.

Four Oliver string discharge primary filters.

Four Northern Foundry string discharger secondary filters. 3 M.P.R., 25″ vacuum. Outer cloth VB-34 clairtwill—21 day life, Padduck cloth, 21oz., 2-3 months.

Two Moore clarifiers. Each with 25, 5′ x 7′ leaves.

Ion Exchange and Precipitation. Of standard design the ion exchange columns have a capacity of 285 cu. ft. each containing IRA resins. Loadings are 4.0-4.5 lbs. U_3O_8 per cu. ft. of resin and salt-sulphuric acid is used as the eluate. The pregnant brine solution is upgraded about five times its feed value. Salt is consumed at the rate of 1.61 lbs. per lb. of U_3O_8 and sulphuric acid at 10 lbs. per ton of ore. This solution is precipitated by magnesium oxide which is wet ground to 90% minus 200 mesh and is manually added. At a pH of 7.0 precipitation is 99.9% and a batch cycle takes about 8 hours.

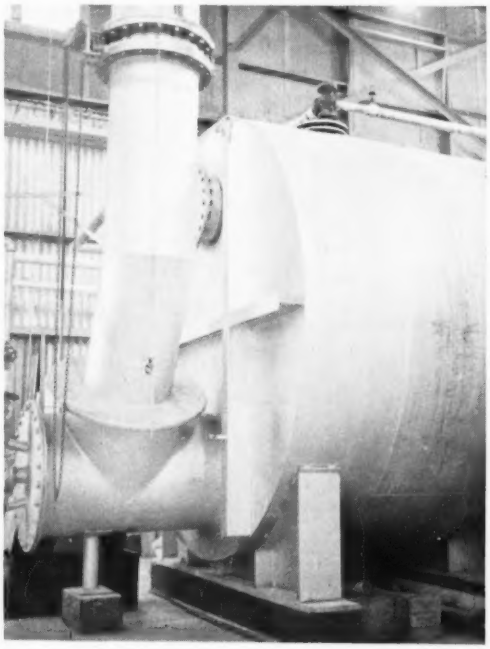
Four column Ion exchange system built to the Permutit Company's standard design. Three column absorption.

Three 20′ x 20′ precipitation tanks each fitted with Greey mixers.

Filtration and Drying. The precipitate is filtered and carefully washed with ¼% sodium sulphate to remove chlorides and each press load is then transferred to a 10ft. Bartlett snow steam dryer. Five to eight hours is necessary to dry 1,200 lbs. of U_3O_8 to less than 10% moisture after which it is packed in drums of 25 Imp. gallons capacity for air transportation to the refinery of Eldorado Mining & Refining Ltd.

Sulphuric Acid

Operating at 25% in excess of their rated daily capacity the two sulphuric acid plants have been producing approximately 200 tons per day of 93% acid. The original unit is a 100 tons per day standard Leonard-Monsanto contact unit which is enclosed and was designed



Sulphur burner at the 100 tons plant.

to meet the original needs of the mill. Following expansion of milling capacity to 1,650 tons daily additional sulphur burning acid plant was installed by the Leonard Construction Company; it is of similar design but has a capacity of 65 tons per day. Molten sulphur for the two units is supplied from a common sulphur melter

which is located in the larger building of the 100 tons acid plant. Elemental sulphur is obtained from the Shell Oil and Royalite

Sulphuric acid installation (100 tons per day left, 65 tons per day right) separated by cooling towers.

Companies gas cleaning operations in Southern Alberta and requirements run at an annual rate of 28,000 to 33,000 tons per annum. The conversion of SO_2 to SO_3 is 97-98% and the cost of acid produced (93% H_2SO_4) is approximately 1 cent per lb.

The favourable operating costs of the plant have been achieved by careful design of plant layout and adoption of modern metallurgical processes. Power requirements of the milling circuits, sulphuric acid and crushing plants, totalling approximately 3,600 H.P., are met from the Company's 6,000 H.P. power plant. Excess heat generated during acid plant reactions is used for mill heating and drying the final uranium product. The operating efficiency of the leaching section is 94.9% (soluble loss 1%) and that of the ion exchange section 99.4%. The plants overall recovery is 91.7% with a metallurgical balance discrepancy of 1.6%.

We are indebted to Mr. A. C. Hoddinott of Gunnar Mines Ltd. for information and illustrations placed at our disposal.

Some aspects of Sulphur Recovery from Industrial Gases in Western Germany

THE utilisation of sulphur contained in industrial gases, notably those of Germany's coal carbonisation industry, is intensive and its momentum is increasing with a view to reducing, if not eliminating, the net cost of gas purification. Impure gas is treated by dry and wet methods, and hydrogen sulphide extracted by the latter means is converted to sulphuric acid and high purity brimstone. It is estimated that sulphur recovery from coal carbonisation, oil refinery and natural gas yields 100,000 tons to 120,000 tons monhydrate annually in the form of 60°Bé acid, as well as over 90,000 tons brimstone, mostly of high purity. In the respective stages of sulphur recovery the following plants and processes are used extensively although not exclusively:—

Desulphurisation by the Alkazid Process.

Among the large number of desulphurisation processes for the removal of hydrogen sulphide and carbon disulphide from industrial gases, the Alkazid Process, on account of its versatility and economic operation is being applied in an increasing number of new fields. Developed by Badische Anilin-and Soda-Fabrik A.G., of Ludwigshafen a/Rhein in co-operation with manufacturers of chemical plant, it is a continuous recycle process, in which gases or liquids are treated in an absorber with Alkazid solutions to remove H_2O and CO_2. The process, which can be carried out at ordinary or high pressure, entails passing gas counter-currently through a bubble cap scrubber at the top of which Alkazid lye is introduced at a temperature of 20°C. The lye selectively extracts at least 90% of the H_2S present in the gas, and only a small percentage of the carbon dioxide. The spent solution is regenerated by heating and then returned to the absorber via the heat exchanger. The stripped gas contains usually about 40% H_2S and 60% CO_2, representing a suitable feed gas for a wet contact sulphuric acid plant or for a combustion in a claus kiln and catalytic recovery of pure brimstone.

The Alkazid process was originally employed in high pressure hydrogenation

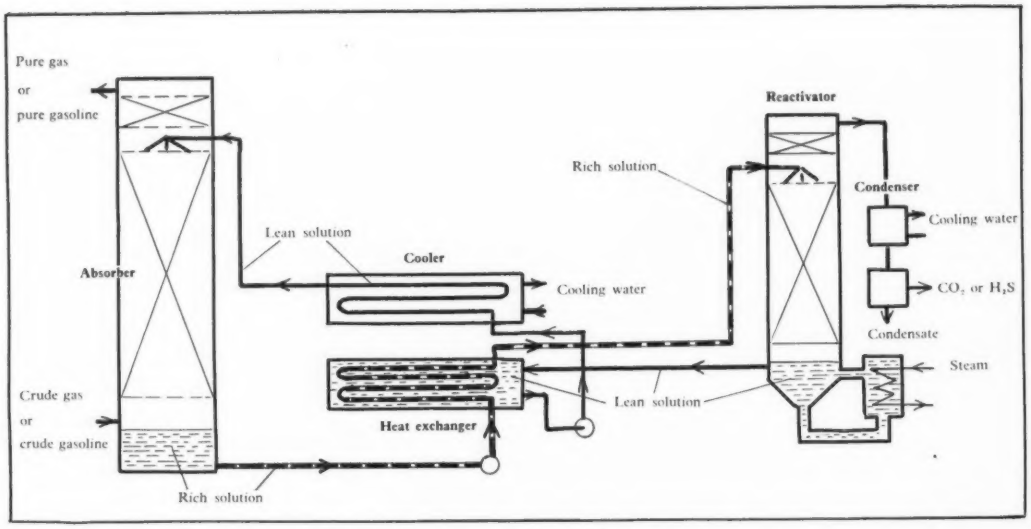


Fig. 6 Flow diagram of the Alkazid process.

plants, Fischer-Tropsch plants and lignite and oil-shale low-temperature carbonisation gas plants. The process has since been applied successfully in the purification of:—

Water gas (coal and lignite)
Natural gas
Petroleum refinery gases
Liquid gases
Converter gas
Flue gas
Platforming gas
Blower gas

The following applications of the Alkazid process and the Alkazid solutions in the removal of H_2S are thought to be of interest.

1. Hydrogenation. The plant has an hourly throughput of 100,000 cubic metres, and gas entering the plant contains 0.25 vol % H_2S. and approx. 8 vol. % CO_2, a ratio of 3/100. After desulphurisation at ordinary pressure the effluent gas from the Alkazid plant contains approx. 0.007 vol. % H_2S. The hydrogen sulphide is recovered in a concentration of 30-40 vol. %, representing an H_2S:CO_2 ratio of 54/100. Selective washing has improved the H_2S/CO_2 ratio 18 fold and the H_2S concentration 140 fold.

2. In the purification of oil-shale low temperature carbonization gases at ordinary pressure and with an initial H_2S and CO_2 content of 20 vol. % and 5 vol. % respectively, a final purity of 0.07 vol. % H_2S is obtained, this represents a scrubbing efficiency of 99.7%. The recovered hydrogen sulphide has a concentration of more than 90%, the rest of the gas consisting of carbon dioxide.

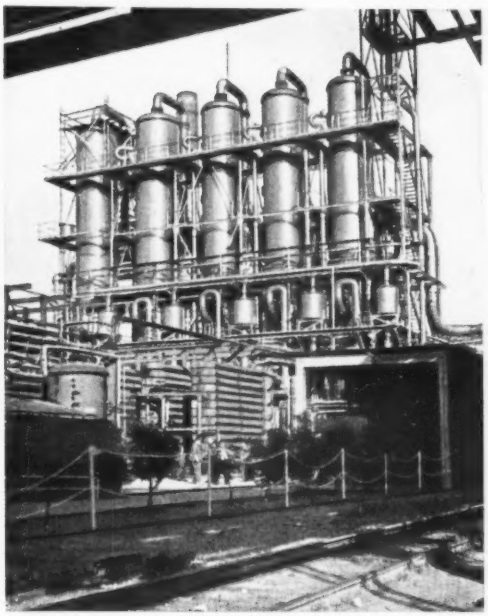
3. Very satisfactory results have been obtained with the Alkazid process in the desulphurization of refinery and hydrogenation gases. In these plants, which operate under pressure, gases containing 10 vol. % H_2S are purified to 0.0013 vol. % H_2S this corresponds to a scrubbing efficiency of 99.99%, despite operation under pressure, only very small amounts of hydro-carbons are washed out.

4. A platforming recycle gas with an H_2S content of 300 mg/cub. m. was purified by washing with Alkazid Solution DIK under pressure to an H_2S content of 15-30 mg/cub. m. (0.009-0.0019 vol. %). This corresponds to a scrubbing efficiency of 90-95%.

Subject to minor fluctuations, dependent on the concentration in the gas of the components to be removed and on the final purity required in the washed gas, the following data serve as indications:

In the scrubbing of gas containing 0.25 vol. % of hydrogen sulphide to a concentration of approx. 0.007 vol. %, approx. 20 kg. of steam are required per kg. of hydrogen sulphide. The scrubbing of hydrogen sulphide from a gas containing 2 vol. % to a final concentration of 0.05 vol. % requires approx.

9 kg. of steam per kg. of hydrogen sulphide. When the hydrogen sulphide concentration is higher the steam consumption is reduced to approx. 4 kg. The figures given refer to plants operating under ordinary pressure for the treatment of gases containing CO_2. The steam consumption is smaller in the case of



Union Rheinische Braunkohlen Kraftstoff A.G., Wesselring. Alkazid desulphurisation plant.

gases which contain no CO_2. The consumption of electrical energy is insignificant in the operation of Alkazid plants. The cooling water consumption varies in accordance with steam consumption, so that in the instances given above approx. 0.5, 0.25 and 0.1 cub. m. of water, respectively, are required per kg. of hydrogen sulphide.

Wet Contact Sulphuric Acid Manufacture

The process and plant developed by Lurgi Chemie, of Frankfurt a. Main, is based on the use of acid gases containing 10% H_2S or more to yield 78% (60% Bé) contact acid without the need to pre-dry the moist SO_2 process gas.

Hydrogen sulphide gas is introduced into a combustion furnace, either by its own pressure or by booster blower (1). Air required for combustion and the subsequent conversion from SO_2 to SO_3 is furnished by blower (3) and passed through filter (2) before entering the combustion furnace. Part of the air stream is diverted to the converter.

The hydrogen sulphide-air mixture, if sufficiently high in calories, burns freely on entering the combustion furnace (4) which consists of a steel shell lined with insulating and refractory brickwork. Additional fuel such as coke oven gas or oil, apart from being required for pre-heating the furnace to combustion temperature, is only necessary where the H_2S-gas is deficient in calories.

Combustion of H_2S to SO_2 takes place at temperatures of 900 to 1,000° C. Since the gas temperature at the converter entry should be between 400 and 450° C., there is excess heat which must be removed. In the case of plants of capacity less than 10 tons of H_2SO_4 60° Bé per day this is done by the radiation cooler (5); in larger units the excess heat may be utilised for generating steam in a waste heat boiler (6). Temperature regulation, where a radiation cooler is employed, is effected by adding air which is drawn off ahead of the combustion furnace. With waste heat boiler installations a by-pass line for the SO_2-gas serves the same end. Apart from being fitted with the necessary ancillary equipment, the waste heat boiler is provided with two feed water pumps, one of them being a steam pump (7) and the other one being electrically driven (8).

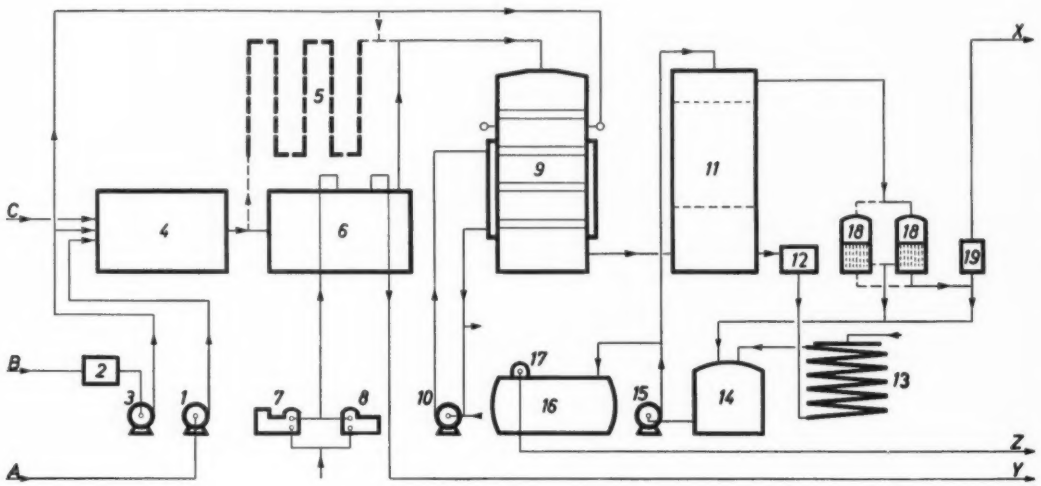
The vanadium pentoxide catalyst which brings about conversion from SO_2 to SO_3 in the converter (9) is distributed over four trays. Being an exothermic process, some of the heat of reaction has to be removed. Small converter units have a cooling jacket through which air is blown by a fan (10); with larger units cold air which has been diverted from ahead of the combustion furnace is introduced into the gas stream after it has passed through the first catalyst bed. Further cooling between the second and fourth catalyst beds is likewise by means of air blown through a cooling jacket, part of the air being circulated to avoid local chilling of the converter shell.

The SO_2 gas leaving the converter at a temperature of approximately 400°C. enters the

Concordia Bergbau A.G., Oberhausen. 10 tons per day wet contact acid plant.

condensation tower (11) which is composed of a steel shell lined with sheet lead and acid-resisting brickwork. Acid produced in the process is circulated through the tower, even distribution being ensured by suitably spaced spray nozzles. The sensible heat of the gas, together with the heat of condensation and dilution, is reduced by passing the acid through water-irrigated coolers (13). On leaving the tower, the acid at a temperature of 70-80° C. flows through the syphon vessel (12) and thence through the tubes of a battery of lead acid coolers. The acid leaving the coolers is collected in the intermediate tank (14) and subsequently recirculated through the tower by means of a pump (15) of acid-resisting material. Part of the acid is discharged from the pump delivery line as product acid into the product acid tank (16). from which the submerged pump (15) delivers it to the acid storage.

Sulphuric acid mist is formed in the condensation tower during cooling of the water-vapour contained in the gas. This acid mist is separated in the ceramic filters (18) followed by a baffle separator (19) to such an extent that only traces of SO_3 are emitted to the atmosphere.



Layout of a LURGI type Wet Contact Plant

A — H_2S B — atmospheric air X — exit gas Y — steam Z — sulphuric acid 60° Bé C — fuel

The resultant wet contact acid is water-white, and the only impurities it contains are approx. 0.1 g N_2O_3/liter, depending on the HCN and NH_3 contents of the H_2S gas. Recently published data indicate that conversion costs of acid gas at a typical wet catalyst sulphuric acid plant with a daily capacity of 10 tons are as follows:—

Power consumption	40 kWh/t 60°Bé acid
Cooling water, 25°C.	30 cu. m./t 60° Bé acid
Conversion of SO_2 to SO_3	in excess of 98%
Steam generation	1.0-1.2 t/t 60° Bé acid
Therefore approx. £ equivalent of operating cost ..	19s. 6d.
Capital cost and interest	23s. 6d.
Total	43s. 0d.
Less Steam credit	19s. 0d.
Net per ton 60° Bé (78%) acid	24s. 0d.

Elemental Sulphur Production by the BASF Claus Process

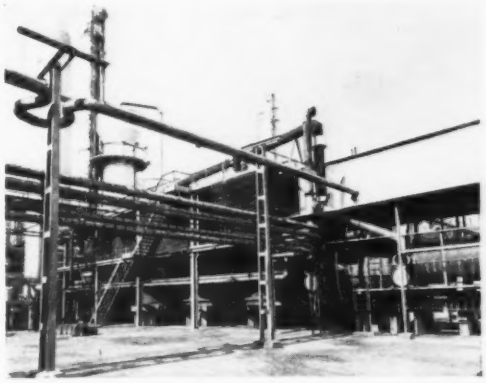
The well-known and widely applied principle of partial oxidation in a Claus kiln of hydrogen sulphide to brimstone and sulphur dioxide, and the fuller reaction of the sulphur dioxide with hydrogen sulphide in a catalytic converter forms the basis of the improved BASF Claus process. These reactions may be expressed as follows:—

(i) $H_2S + 1\frac{1}{2}0_2 \longrightarrow SO_2 + H_2O + 124$ Cal
(ii) $H_2S + \frac{1}{2}0_2 \longrightarrow S + H_2O + 53$ Cal
(iii) $SO_2 + 2H_2S \longrightarrow 3S + 2H_2O + 35$ Cal

The partial combustion of the hydrogen sulphide carried out in a specially designed Claus kiln gives rise to the production of steam. The combustion temperature in the kiln is carefully controlled by adjustment of the entering gas and air proportions so that maximum conversion efficiency is obtained in the catalytic converter.

The further reaction of sulphur dioxide with hydrogen sulphide is carried out in two stages, the gases being cooled after leaving the converter to condense the sulphur formed. By this means the forward reaction, ie., the formation of sulphur, is promoted according to equation (iii). The gas leaving the first catalytic converter is cooled with liquid sulphur so that sulphur contained in it is removed. After removal of sulphur, the gas is reheated before entering the second catalytic converter, the gas leaving the second catalytic converter is similarly cooled and scrubbed with liquid sulphur to remove sulphur. The effluent gas is passed through a final converter where residual hydrogen sulphide is oxidised to sulphur dioxide. BASF catalyst is used in the Claus catalytic converters.

The BASF Claus process can be employed for the recovery of sulphur from gases with a hydrogen sulphide content of 10% by volume



Svenska Sifferolje A.B., Närkes Kvarntorp. One of the four BASF Claus kilns. Alkazid stripping tower left.

and more. The yield of elemental sulphur normally lies between 80 and 94%, based on the hydrogen sulphide content of the gas. The heat removed in the waste heat boiler is used to generate steam which may be used to heat the molten sulphur lines and storage tank. In the BASF Claus plant approximately two tons of steam is produced for each ton of sulphur; this is usually in excess of sulphur plant requirements, and steam becomes available for use elsewhere. Power requirements for the air blower and the sulphur circulation pumps are about 50 KWH per ton of sulphur in the case of large-scale plants.

We are indebted to Messrs. Badische Anilin-& Soda-Fabrik A.G. and Lurgi für Chemie und Hüttenwesen m.b.H. for information and illustrations placed at our disposal and acknowledge data published in Glückauf of 27.4.57.

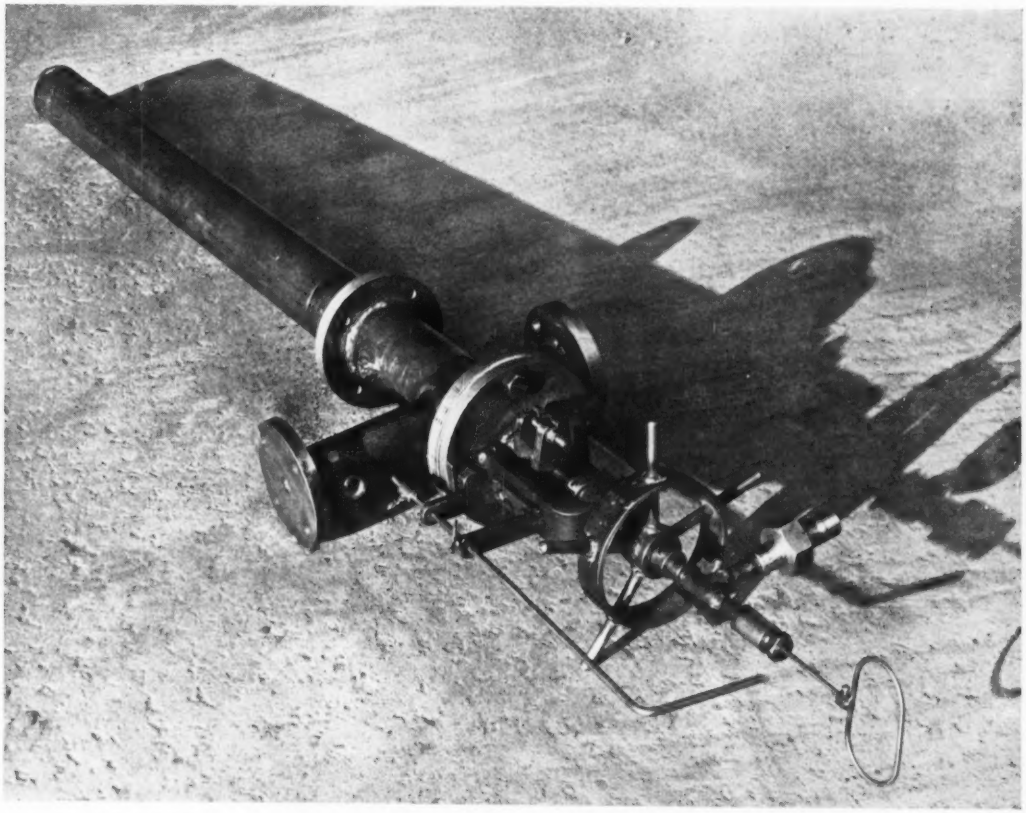
Conversion of Pyrites Roasters for Burning Elemental Sulphur

NOW that sulphur is plentiful again after the shortage of the early 1950's, sulphuric acid manufacturers especially in the United Kingdom have been looking into the question of converting their pyrites-burning plants to burn sulphur. The advantages of a flexible system whereby a changeover can be quickly and inexpensively adapted from pyrites-burning to sulphur-burning or vice-versa, are evident, especially as much of the uncertainty about the future availability of raw materials is removed and short term fluctuations of raw materials prices can be exploited.

Plant modification is most easily effected if the sulphur is burned in the roaster of a "converted" pyrites-burning plant and the sulphur dioxide-containing gases produced pass through the gas purification system. Since cold gas purification is retained, hot gas purification equipment is not essential and sections of it may in most cases be by-passed without detriment to the subsequent sections of the plant.

If the roaster is followed by a waste heat boiler, heat can be recovered when burning sulphur, although the quantity is less than when burning pyrites because the heat of combustion per unit weight of sulphur is lower. The gas leaving the drying tower is pre-heated to the required temperature for entering the first stage of the converter in the same SO_2/SO_3 heat exchangers as are used when burning pyrites and no modification at all to the oxidation system is required.

Most of the pyrites-burning plants built in Britain during the early 1950's are of the



James Spray Burner.

flash roasting type, and the conversion of a flash roaster for the burning of elemental sulphur is a simple process if carried out in the way it was done at one large plant in this country earlier this year. On this installation a James spray burner or "gun" designed by Imperial Chemical Industries Ltd., and licensed to Simon-Carves Ltd., has been mounted in the side of the bottom cone of the roaster. Molten sulphur, in that particular case from an existing melter, is pumped at a pressure of up to 40 lb./sq. in. to the steam-jacketed spray burner, where it is atomised by compressed air at a slightly lower pressure. With several burners it would be advisable to feed the burners from a molten sulphur ring main. The roaster is pre-heated to about 500-600°C by means of an oil burner before feeding sulphur to it.

The sulphur gun is capable of operating over a wide range of sulphur input—approximately 10-40 tons per day—and the strength of the gas leaving the roaster can be maintained at a steady value anywhere in the range 7-12% SO_2. Filtration of the sulphur fed to a James gun is quite unnecessary with most of the grades at present being used in this country, which include high purity recovered sulphur, bright and off-colour U.S. Frasch sulphur and filtered and mixed Mexican off-colour Frasch sulphur.

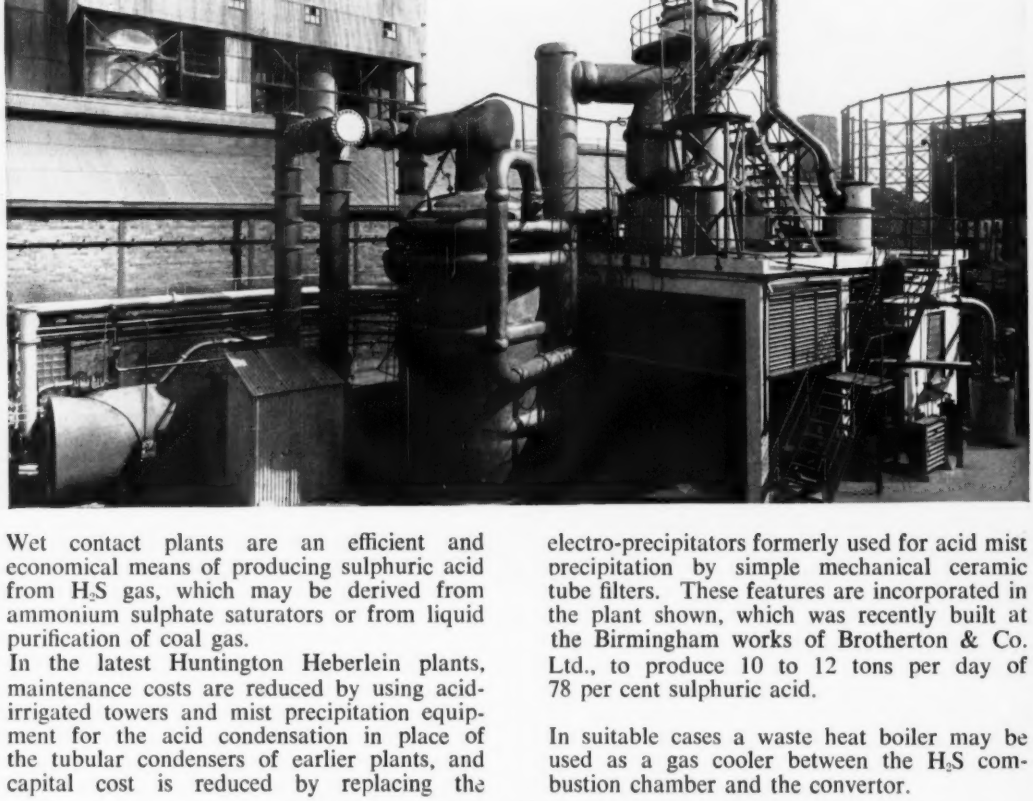
Other measures to obtain flexibility in the use of sulphurous raw materials include the installation of a Glens Falls sulphur burner as an alternative to existing pyrites roaster, while employing the same converter section.

Solid sulphur has also been burned successfully in flash roasters and may be burned in a fluidised bed roaster by mixing it with sand and feeding it in the same way as pyrites.

We are indebted to Messrs. Simon-Carves Ltd. for information and photograph placed at our disposal.

WET CONTACT PLANTS

for sulphuric acid production from hydrogen sulphide



Wet contact plants are an efficient and economical means of producing sulphuric acid from H_2S gas, which may be derived from ammonium sulphate saturators or from liquid purification of coal gas.

In the latest Huntington Heberlein plants, maintenance costs are reduced by using acid-irrigated towers and mist precipitation equipment for the acid condensation in place of the tubular condensers of earlier plants, and capital cost is reduced by replacing the electro-precipitators formerly used for acid mist precipitation by simple mechanical ceramic tube filters. These features are incorporated in the plant shown, which was recently built at the Birmingham works of Brotherton & Co. Ltd., to produce 10 to 12 tons per day of 78 per cent sulphuric acid.

In suitable cases a waste heat boiler may be used as a gas cooler between the H_2S combustion chamber and the convertor.

Wet contact sulphuric acid plants by

Huntington, Heberlein & Co Ltd

SIMON ENGINEERING GROUP

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